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Annual report 2015 of the EURL-FCM

on activities carried out for the implementation of Regulation (EC) no 882/2004

EURL
European Union Reference Laboratory
for Food Contact Materials

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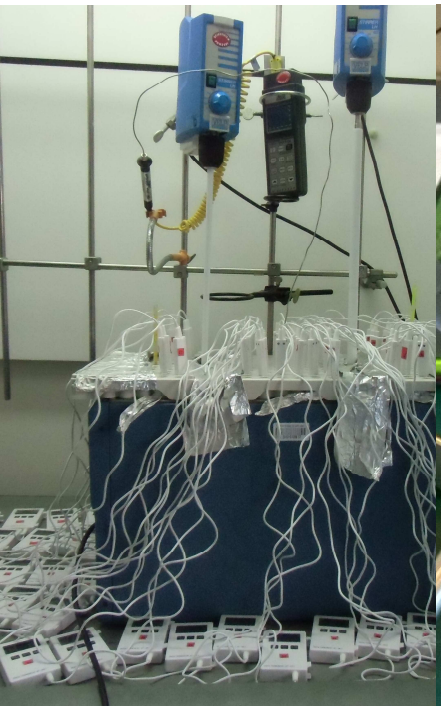

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Abstract

This annual report presents the activities of the EURL FCM in 2015 under the Administrative Agreement N° SI2.701410.

The work programme for 2015 was established on the basis of the priorities established together with DG SANTE in conformity with the objectives and priorities laid down in the Commission Implementing decision C(2015) 4993 and the financial contribution to the European Union reference laboratories. The work programme aims to contribute to a high level of health, ensuring a high level of protection for consumers and the environment, while favouring competitiveness and creation of jobs. It responds to the specific objectives to contribute to a high level of safety of food and to improve effectiveness, efficiency and reliability of official controls. The layout of this report and its deliverables follow the operational objectives as laid down in the Annex of the Commission Implementation Decision, as follows: 1) to ensure the development and use of high quality analytical methods across the EURL framework 2) to maintain appropriate level of proficiency testing (PT) ensuring efficiency of control analysis methods and 3) to ensure the availability of scientific and technical assistance provided by the EURLs.

Under Objective 1, the deliverables included 1) method descriptions on the identification of polymers in multilayers as follow up to the proficiency testing of 2014, 2) an investigation on recyclability of simulant E to improve cost effectiveness of testing regimes using the simulant for dry foods, 3) a compilation of NRL methods, and 4) collaboration with third countries.

Under objective 2, the deliverables included 1) one resulting method as follow-up PT on quantification of metals from plastics 2) one PT the optimisation of temperature control in migration testing protocols, 3) a multianalyte interlaboratory comparisons (ILC) on specific migration testing and 4) potential follow-up actions on National Reference Laboratories (NRLs) performance.

Under Objective 3, the deliverables included 2 workshops (on ceramics and on latest development in research for FCMs). The plenary sessions included a forum on the planning the update to the guidance on kitchenware. Deliverables also included provision of expertise to Commission, member states, NRLs and EFSA, networking activities, external presentations, as well as reciprocal exchange of information with professional bodies and stakeholders.

A set of specific outputs were generated to respond to the specific priority on development and comparison of testing regimes for metals migration from ceramic materials in the context of the revision of Council Directive 84/500/EEC. These included 1) a report on precision criteria for multi elements for metals from ceramics by inductively coupled plasma mass spectrometry (ICP-MS), optical emission spectrometry (OES), atomic absorption spectroscopy (AAS), 2) a report on scoping studies on testing regimes for rim, 3) a report on investigation on tests to simulate the use of crystalware, and 4) a report on comparisons of impact of testing regimes for ceramics and preliminary results for bakeware.

In addition, continuing work items expressed as specific priority included the development of a repository for substances regulated under the Regulation 10/2011 for plastics as well as the development of an inventory of methods of analysis.

This report also includes performance indicators comparing the achieved with the planned outcomes and deliverables. The EURL FCM achieved the ex-ante objectives and delivered as planned.

1. Introduction

The European Commission Joint Research Centre (JRC) was established in 2004 by the Official Feed and Food Controls Regulation EC No 882/2004 to act as the European Union Reference Laboratory for Food Contact Materials (EURL FCM). It is hosted in the Chemical Assessment and Testing Unit of the Institute for Health and Consumer Protection. The unit has currently 44 staff members of which 7 were in 2015 executing tasks primarily linked to the EURL and related food contact activities for DG SANTE.

In line with the JRC's mission statement, different units in the JRC provide scientific and technical support to policy development and implementation of the EU's regulatory framework for Food Contact Materials (FCMs). The policy support includes amongst others the operation of the EURL-FCM. Its mandate is implemented by an administrative arrangement (AA) between DG SANTE and the JRC. The JRC covers some activities of the EURL-FCM from its own budget along with the financial support from the AA.

The EURL FCM contributes to the implementation of the OFFC Regulation through development, validation of methods and proficiency testing for migration from FCMs, provision of methods to NRLs, and by coordinating the EU network of FCM National Reference Laboratories (NRLs), ensuring its consistent performance. The EU network of FCM Laboratories includes NRLs throughout the EU, as well as Switzerland and Norway. Currently NRL-Malta is represented by NRL-UK, NRL-NO is represented by NRL-DK.

This report represents the summary of the deliverables under the Administrative Agreement N °SI2.701410 and covers all activities of the EURL, co-financed or not by this AA.

2. OPERATIONAL OBJECTIVE 1 –Development and use of high quality analytical methods

The mandate of the Commission Implementation Decision includes the following aspects:

- *Ensuring dissemination of analytical and reference methods from EU-RLs to NRLs;*
- *Monitoring of publication by EU-RLs of new developed methods and corresponding validation studies;*
- *Coordination of EU-RL activities on practical arrangements for the application of new analytical methods;*
- *EU-RLs, NRLs and Member States coordination for the preparation of the 2016 work programmes regarding new or improved methods, and/or dissemination information means on methods and reference materials;*
- *planning of trainings, meetings and workshops organised by the EU-RLs for the harmonization of diagnostic techniques and of methods of analysis;*
- *Initiation of EU-RLs collaboration with laboratories in third countries.*

2.1 Repository of references substances for FCM

This work focused on the expansion (continuous) of commercial and /or analytical sources of substances for FCM according to the latest amendments of Regulation 10/2011 and the submission of dossiers by petitioners to EFSA. The work also included to prepare and send upon request standard calibrants not available commercially to NRLs (if present in the reference collections) and other stakeholders for use for research or enforcement purposes under approved programmes.

In 2015 JRC received from petitioners representative small samples for five substances. The EURL-FCM received 6 requests from stakeholders for aliquots to use as calibrants of substances that were held in the repository and provided these substances timely.

2.2 Guidance document on test methods for the identification of polymers

Being able to identify the composition of a packaging film is essential to confirm the correctness of specifications given in the Declaration of Compliance that accompanies all FCMs made of plastic (see EU 10/2011 Art. 15 and Annex IV (3) [1]). Each type of polymer, printing, coating and adhesive contains typical additives and monomers or oligomers with a potential for migration, while at the same time certain layers can act as functional barriers for particular migrants. Therefore, knowing the composition of a multilayer film enables laboratories to predict which migrants can be expected from the packaging film and thus also helps to check the plausibility of obtained migration results.

A PT was organised in 2013 which for the first time aimed at estimating performance of qualitative analysis. The aim of the exercise was to test the laboratories' ability to identify unknown plastics materials via quick screening tools. The results demonstrated a spread and some level of difficulty for laboratories to identifying particular multilayer materials. A follow up was organised in 2014 specifically for multilayers, in order to ensure improved laboratory performance.

Considering there are no reference methods and the novelty to tackle assessments of qualitative nature, EURL produced a number of methods descriptions to assist NRLs in the doing this type of analysis. The descriptions have now been compiled into a final guidance document.

Report:

Guidance for the identification of polymers in multilayer films used in food contact materials: User guide of selected practices to determine the nature of layers

Anja Mieth, Eddo Hoekstra, Catherine Simoneau

Report EUR 27816 EN, JRC100835 ISBN 978-92-79-57561-7 (PDF), ISSN 1831-9424 (online), doi:10.2788/10593 Luxembourg: Publications Office of the European Union, 2015

Abstract

This guidance describes how to characterize the composition of a multilayer plastic film for food packaging, with respect to the consecutive order of the layers and their identity. It provides necessary background information on the general composition of multilayer plastic packaging and it illustrates in detail the separation of layers for some examples. It also provides in annexes additional information related to the use of a microtome and of optical microscopy using one common instrument for illustrative purposes.

2.3 Cost effectiveness for optimum performance of testing with food simulant E

Regulation (EU) No 10/2011 established poly (2,6-diphenyl-p-phenylene oxide) or PPPO, commercially known as Tenax®, as a food simulant E for testing specific migration into dry foodstuffs. A validation of method took place in 2012 and a first time PT was

organised in 2013, with a follow-up in 2014. PPPO is a very costly consumable averaging 200 € for 10g, and one test usually consumes at least 10g per sample. Thus being able to recycle this simulant would greatly increase the cost effectiveness of testing and be of benefit to all member states. However, this cost effectiveness cannot come at the detriment of a reduced performance of the test. Therefore one aspect that had not been investigated neither in academic studies nor at commercial level was whether the regeneration of Tenax® may be feasible, and in which case how many cleaning cycles a given test batch of Tenax® can withstand before the migration results are no longer comparable. It has also been noted by the EURL and several NRLs that batches of the same specification may be delivered with different appearances. Specifications on pore size etc. are included in the Regulation for historical reasons but it is unknown whether these qualities change with use and make the technical specifications not amenable to be respected in practice.

The main objective of this work was to assess the effect of recycling PPPO. When used as dry food simulant for testing specific migration, PPPO (grade particle size 60-80, mesh and pore size 200 nm) must be cleaned prior to its use according a procedure described in the CEN method EN 14338:2004[1]. This method consists of performing a Soxhlet procedure for 6 hours using acetone as solvent. Afterwards PPPO is dried in an oven at 160 °C during 6 h. As this method has been adequate to clean PPPO, a common practice carried out in laboratories has been to reuse the same PPPO portion in different migration assays after applying the cleaning procedure. No data is available the performance of PPPO when reused in migration tests after cycles of cleaning. One hypothesis would be that reuse could modify the particle size of PPPO and therefore this could have an impact on the results obtained in migration testing.

Approach

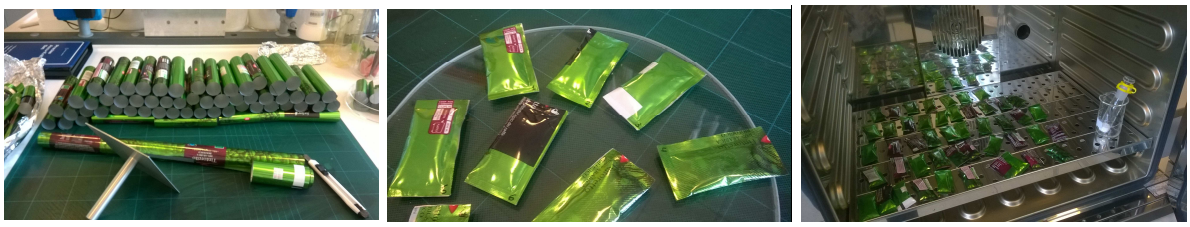
A portion of virgin PPPO of approximately 50 g was taken. This portion was cleaned once by Soxhlet with acetone during 6 h. PPPO was then put in a Petri dish and left under the fume hood for acetone evaporation. Then, PPPO was placed in the oven at 160 ºC for 6 h. This was considered the first Soxhlet cycle. After this procedure PPPO was ready to be used in migration tests. A multilayer film composed of polyethylene terephthalate (PET), aluminium (Al) and polyethylene (PE) and used as packaging for coffee was used as standard test material for the migration tests. The film contained C11, C12, C13, C17, C16, C20, C22[2], LAC1 and LAC2, DBS, DEHA, and Erucamide). An assessment was performed of the homogeneity of the chemicals in the packaging material.

Homogeneity studies

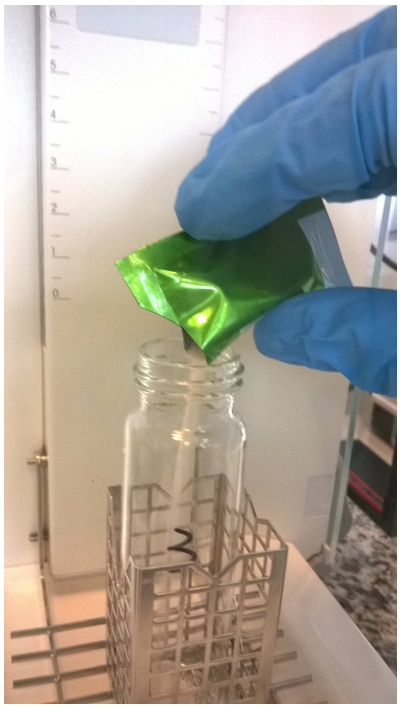
[1] EN 14338:2004 - Paper And Board Intended To Come Into Contact With Foodstuffs - Conditions For Determination Of Migration From Paper And Board Using Modified Polyphenylene Oxide (mppo) As A Simulant

[2] C11, C12, C13, C17, C16, C20, C22: Alkanes chains. LAC1 and LAC2: lactones; DBS: dibutyl sebacate; DEHA: de ethyl hexyl adipate.

Preliminary studies were also conducted to define the time and temperature of contact between packaging and food simulant E (PPPO) for the migration tests. The contact condition was chosen according Reg. 10/2011 for specific conditions for contact times above 30 days at room temperature, where migration tests must be performed for 10 days at 60 °C. In order to accelerate testing time and temperature conditions the Arrhenius formula was applied (t2 = t1 * Exp ((-Ea/R) * (1/T1-1/T2)), which gave accelerated tests of 1 day at 90 ºC. Migration tests were conducted on pouches of 0.25 dm^2 filled with ca. 1g of PPPO and exposed (1 day 90 ºC).



Pouches made and used in the studies



Following the exposure, the PPPO containing the migrated substances was extracted with hexane and analysed by GC/MS. The performance characteristics of the method to determine migrants from PPPO (linearity, precision, recovery, matrix effect) were evaluated. The PPPO was then cleaned by Soxhlet and assessed for its physical characteristics. The particle size distribution on the PPPO was assessed using sieves (ASTM certified of 20, 35, 60, 80 and 100 mesh) in a vibratory shaker after each or several cycles of Soxhlet. The PPPO particle shape was also analysed by optical microscopy.

Eleven migration tests were carried out using the same PPPO. The first migration test was performed with 46 pouches and in the consecutive migration tests until the last one, three pouches were analysed. In every test a blank (ca. 1 g PPPO in a glass tube) was analysed in same conditions. A multianalyte solution containing all the standards (C11, C12, C13, C17, DBS, DEHA, Erucamide) was used for quantification. Calibration with multianalytes was done in PPPO and with the same extraction procedure as the test samples.

Migration values

Migration values obtained after exposing to the film the same PPPO that had been recycled are detailed in **Table 1** and **Fig. 1.** The values are expressed as mg of analyte per kg of PPPO, and also in mg of analyte per kg of packaging.

Table 1

Migration values versus number of migration assays and cycles of cleaning (mg analyte per kg PPPO) ± SD

Analyte	1st Migration (*n*=47)	2nd Migration (*n*=43)	3rd Migration (*n*=40)	4th Migration (*n*=37)	5th Migration (*n*=34)	6th Migration (*n*=31)	7th Migration (*n*=28)	8th Migration (*n*=25)	9th Migration (*n*=22)	10th Migration (*n*=16)	11th Migration (*n*=14)
	1st Soxhlet	2nd Soxhlet	3rd Soxhlet	4th Soxhlet	7th Soxhlet	10th Soxhlet	15th Soxhlet	20th Soxhlet	25th Soxhlet	35th Soxhlet	45th Soxhlet
C11	2.25±0.46	2.39±0.23	2.44±0.14	2.26±0.13	2.15±0.14	2.02±0.11	2.21±0.11	2.04±0.09	1.69±0.07	1.47±0.13 (*n*=14)	1.09±0.07
C12	5.41±0.54	5.12±0.39	5.28±0.28	5.15±0.25	5.17±0.24	5.05±0.24	5.03±0.26	5.03±0.24	4.69±0.24	4.11±0.30 (*n*=14)	3.60±0.30
C13	5.45±0.24	5.17±0.27	5.27±0.19	5.22±0.18	5.30±0.20	5.24±0.24	5.18±0.20	5.25±0.23	5.02±0.21	4.35±0.20 (*n*=14)	4.21±0.17
C16	1.83±0.05	1.78±0.06	1.79±0.05	1.77±0.05	1.84±0.06	1.84±0.06	1.82±0.05	1.76±0.06	1.79±0.06	1.47±0.06	1.40±0.04
C18	2.25±0.06	2.16±0.08	2.19±0.07	2.16±0.06	2.26±0.08	2.26±0.08	2.25±0.07	2.19±0.07	2.08±0.06	1.71±0.08	1.65±0.04
C20	2.52±0.08	2.37±0.09	2.40±0.08	2.37±0.07	2.51±0.09	2.52±0.09	2.51±0.08	2.42±0.09	2.37±0.07	1.96±0.09	1.96±0.04
C22	2.67±0.08	2.57±0.10	2.50±0.09	2.59±0.09	2.57±0.10	2.58±0.08	2.51±0.10	2.47±0.12	2.46±0.07	2.00±0.11	2.21±0.08
ADP	3.93±0.12	3.29±0.14	3.44±0.09	3.35±0.06	3.89±0.10	3.92±0.10	3.64±0.12	2.87±0.06	2.82±0.07	2.24±0.08	1.83±0.05
LAC1	2.80±0.07	2.75±0.10	2.80±0.09	2.76±0.05	2.69±0.10	2.67±0.08	2.76±0.08	2.89±0.06	2.69±0.07	2.43±0.08	1.93±0.05
LAC2	0.77±0.04	0.64±0.03	0.65±0.02	0.66±0.02	0.76±0.02	0.77±0.02	0.70±0.04	0.68±0.05	0.70±0.05	0.48±0.03	0.45±0.01
DBS	3.48±0.87	1.89±0.39	1.84±0.47	2.02±0.44	2.42±0.40	2.57±0.48	2.61±0.82	1.35±0.52	1.90±0.64	1.32±0.43	1.08±0.34
ERU	50.07±4.31	42.17±4.63	37.10±3.57	41.93±3.57	40.49±3.89	39.56±3.67	40.59±4.10	30.43±4.16	44.92±4.68	30.98±2.35	16.88±3.13
	Same migration values expressed as mg analyte per kg film ± SD										
C11	7.73±1.55	8.03±0.79	8.14±0.46	7.61±0.46	7.29±0.46	6.82±0.32	7.60±0.42	7.04±0.32	5.82±0.26	5.15±0.45 (*n*=14)	4.44±0.27
C12	18.58±1.81	17.23±1.45	17.63±0.97	17.38±0.93	17.51±0.78	17.08±0.72	17.33±0.85	17.45±0.80	16.18±0.80	14.41±0.97 (*n*=14)	14.61±1.19
C13	18.71±0.82	17.39±1.02	17.59±0.69	17.61±0.66	17.95±0.59	17.71±0.69	17.84±0.56	18.21±0.69	17.35±0.72	15.24±0.57 (*n*=14)	17.08±0.71
C16	6.27±0.14	5.97±0.18	5.97±0.17	5.96±0.14	6.22±0.14	6.21±0.19	6.26±0.17	6.10±0.15	6.18±0.17	5.14±0.13	5.67±0.20
C18	7.73±0.18	7.28±0.23	7.30±0.24	7.28±0.19	7.66±0.19	7.63±0.22	7.74±0.23	7.61±0.19	7.18±0.20 20	5.97±0.16	6.71±0.22
C20	8.65±0.26	7.96±0.25	8.00±0.28	7.99±0.23	8.50±0.21	8.53±0.26	8.64±0.24	8.38±0.24	8.19±0.22	6.85±0.21	7.96±0.25
C22	9.18±0.20	8.64±0.27	8.36±0.33	8.73±0.25	8.69±0.26	8.72±0.24	8.64±0.32	8.56±0.35	8.51±0.23	6.99±0.27	8.97±0.41
ADP	13.48±0.36	11.05±0.51	11.49±0.31	11.29±0.27	13.15±0.25	13.26±0.28	12.52±0.44	9.96±0.25	9.74±0.24	7.83±0.32	7.43±0.19
LAC1	9.63±0.22	9.25±0.39	9.33±0.32	9.31±0.20	9.09±0.21	9.04±0.30	9.49±0.24	10.01±0.28	9.28±0.24	8.49±0.31	7.84±0.22
LAC2	2.65±0.12	2.15±0.10	2.18±0.08	2.23±0.05	2.56±0.07	2.62±0.08	2.40±0.12	2.36±0.15	2.42±0.16	1.69±0.12	1.81±0.05
DBS	11.93±2.97	6.36±1.29	6.14±1.59	6.79±1.42	8.21±1.43	8.69±1.60	8.98±2.81	4.67±1.81	6.55±2.17	4.61±1.57	4.38±1.43
ERU	171.97±14.80	141.73±15.15	123.90±12.13	141.51±12.21	137.10±13.73	133.75±11.86	139.71±12.95	105.40±13.77	155.13±16.31	108.15±8.55	68.55±12.79

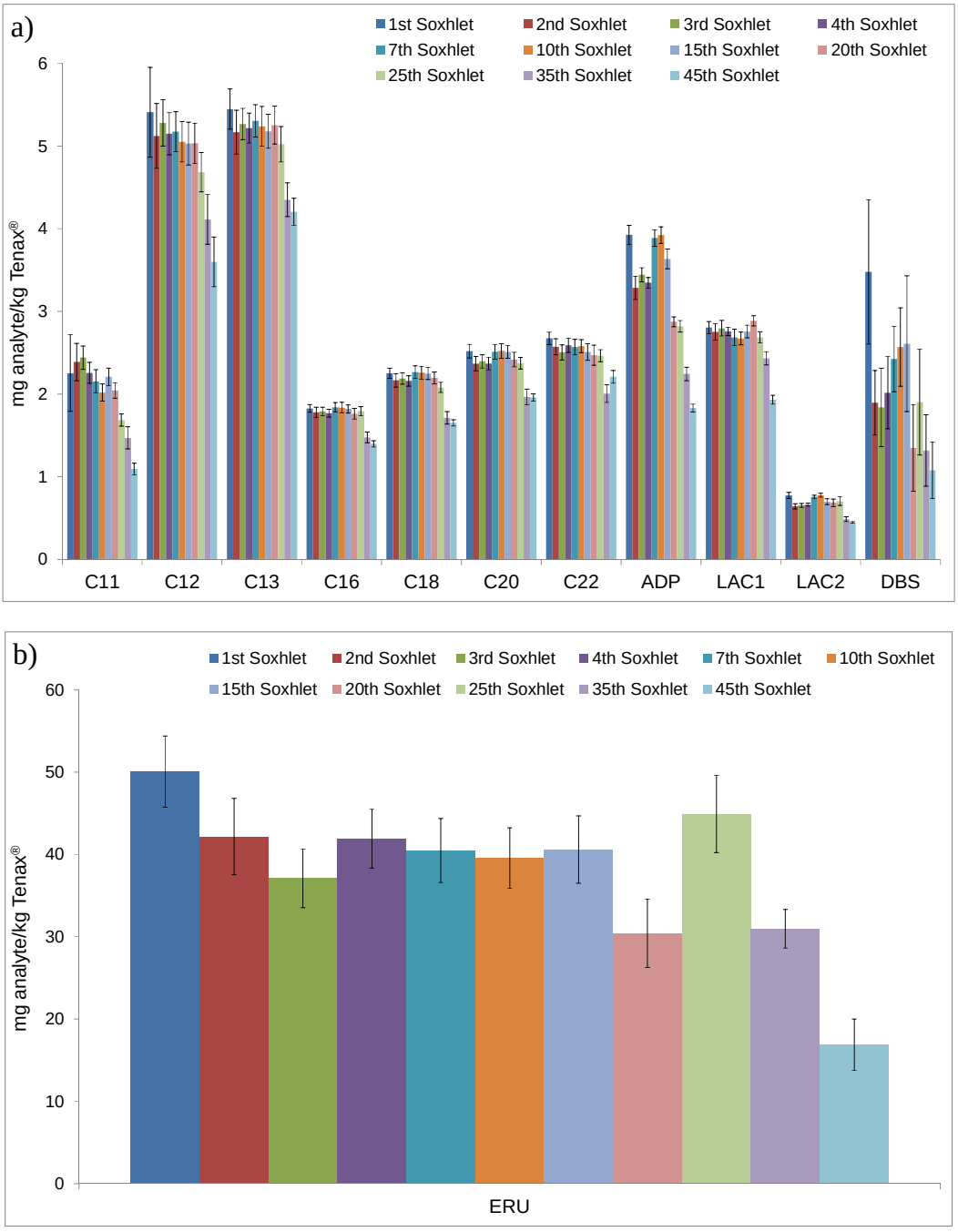


Figure 1. Overview of migration levels obtained after eleven migration tests and several cycles of cleaning using same portion of PPPO (a) most of the compounds and (b) erucamide.

In order to verify differences in the migration values after several migration tests and cycles of Soxhlet performed, the approach used to check the stability of reference materials in proficiency testing was applied (ISO GUIDE 35:2006[3]). The evaluation of data was carried out by performing a linear regression on the concentration of the migrants (mean value obtained from each migration test) versus the number of Soxhlets performed. The evaluation was also carried out taking the number of migration tests into account. For a stable material, it is expected that the intercept is (within uncertainty)

[3] ISO Guide 35:2006 - Reference materials -- General and statistical principles for certification

equal to the assigned value which in this case is the migration value, whereas the slope does not differ significantly from zero. The stability results are given in **Table 2**. Migration levels of substances such as C16, C20, C22, LAC1 and LAC2 were stable during all the experiments, i.e., up to the 11th migration test, after PPPO had been recycled for 45 times. For the other substances such as C18, DBS and ERU the migration was considered stable until the 35th cycle of Soxhlet and the 10th migration test. The migration of ADP and C13 remained the same up to the 25th cleaning cycle and the 9th migration test. Finally, for C11 and C12 the stability was not altered up to the 15th (7th migration) and 7th (5th migration) cycle of Soxhlet, respectively.

Table 2: Stability

Stability up to 45 cycles of Soxhlet and 11 migration tests					
Substance	*Intercept*	*Slope*	*s(b1)*	*$t(\alpha=0.95,n-2)\cdot s(b1)$*	*$\lvert b1\rvert < t(\alpha=0.95,n-2)\cdot s(b1)$*
C11	2.4161	-0.0274	0.0026	0.0060	Not stable
C12	5.4192	-0.0357	0.0038	0.0085	Not stable
C13	5.4364	-0.0248	0.0039	0.0088	Not stable
C16	1.8672	-0.0088	0.0092	0.0207	Stable
C18	2.3022	-0.0129	0.0024	0.0055	Not stable
C20	2.5273	-0.0114	0.0065	0.0148	Stable
C22	2.6400	-0.0114	0.0089	0.0202	Stable
ADP	3.8419	-0.0422	0.0073	0.0164	Not stable
LAC1	2.8719	-0.0146	0.0164	0.0372	Stable
LAC2	0.7454	-0.0056	0.0619	0.1400	Stable
DBS	2.5342	-0.0324	0.0155	0.0351	Stable
ERU	45.0012	-0.4784	0.1255	0.2839	Not stable
Values of stable results for the following substances					
C11	2.3455	-0.0168	0.0100	0.0258	Stable (up to 15th Soxhlet)
C12	5.3156	-0.0260	0.0198	0.0631	Stable (up to 7th Soxhlet)
C13	5.3158	-0.0085	0.0046	0.0109	Stable (up to 25th Soxhlet)
C18	2.2272	-0.0028	0.0029	0.0070	Stable (up to 25th Soxhlet)
ADP	3.7530	-0.0304	0.0175	0.0414	Stable (up to 25th Soxhlet)
ERU	43.3967	-0.2929	0.1522	0.3509	Stable (up to 35th Soxhlet)

These results show that PPPO should not be reused too extensively in the same manner for different classes of substances. In addition, this study demonstrated that more attention should be paid regarding the reuse of PPPO as a food simulant. Indeed, the declaration of compliance of packages and articles for food contact may be based on migration values obtained with PPPO. If the test might have used extensively recycled PPPO, an impact on the results will be expected which may influence the judgment of compliance. Further analyses using reference packaging materials and model chemical substances to cover different polarity and functional groups could be useful in order to produce a more general guideline or standard operation procedure for migration assessment with food simulant E including recommendations such as quantification of migrants using a matrix-matched calibration and how many times PPPO could be reused.

Particle size and particle shape

Sieve analyses were performed in order to verify whether the cycles of cleaning or the migration tests followed by the extraction could affect the particle size of the reused PPPO. As can be seen in **Table 3** and **Fig. 2** PPPO obtained after 1st and 45th cycles of Soxhlet presented 88% and 90% of particles distributed between 60 and 80 mesh (180-

250 μm), respectively. This distribution was similar to that declared by the producer in the certificate of analysis for this batch of PPPO. There was a suspicion that after reusing the same portion of PPPO several times a significant decrease in the particle size would occur. However the results suggested that no major changes occurred in the particle size even after 11 migration tests and 45 cycles of Soxhlet performed on the same PPPO portion. However whereas after one cycle of Soxhlet 9% of the particles were in the range of 250 μm, this percentage was reduced to ca. 1% after 45 cleaning cycles. This same behaviour was also observed after 20-35 cycles of cleaning but in this case only 1.5 g of PPPO was used during sieving and the standard deviation was higher than when 10 g was used during analysis. Additionally, differences were observed with respect to the particle sizes smaller than 150 and greater than 250 μm, respectively. In the certificate of analysis there were no reported particles higher than 250 μm and smaller than 150 μm but this study quantified ca. 2-3% of PPPO distributed in these levels after the 1st and the 45th cycles of Soxhlet.

Table 3: Particle size distribution

Mesh	Micron	Soxhlet cycle (% of particle distribution)					COA batch*
		1st	20th	25th	35th	45th	
>100	<150	2.4	2.7	4.0	6.6	2.5	0
80/100	150	7.6	12.0	11.3	15.3	7.6	5
60/80	180	78.9	81.9	81.1	75.8	88.8	86
35/60	250	9.0	2.0	1.3	1.3	1.1	9
20/35	500	1.6	0	0	0	0	0
<20	800	0	0	0	0	0	0

* Certificate of analysis

60-80 mesh after 1st Soxhlet (upper image) vs. after 45th Soxlhet (lower image)

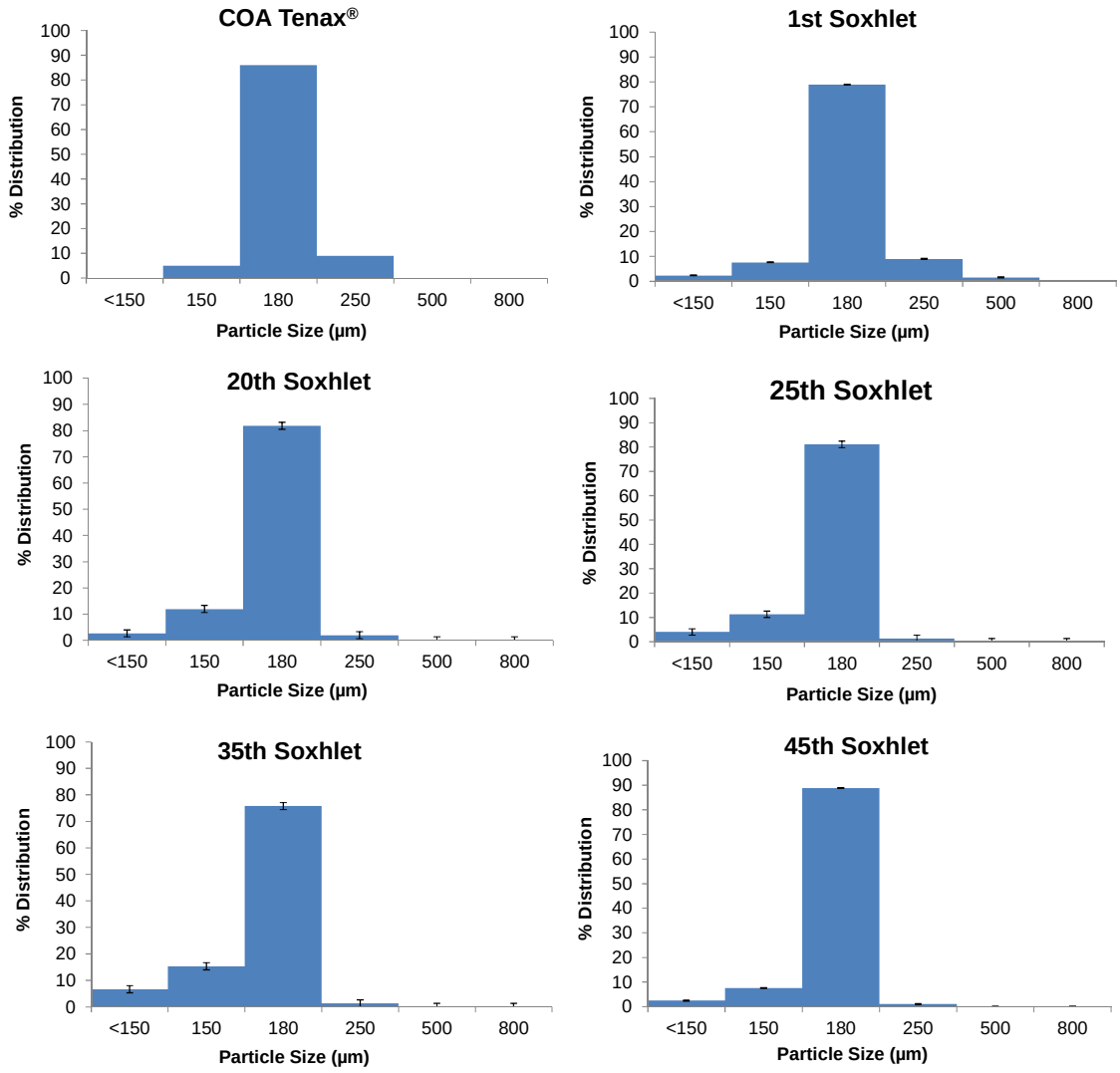


Figure 2. Histogram of the individual fractions of PPPO distributed between <150 and 800 µm.

Conclusions

This work item represents the first research carried out at EU level on the evaluation of the reuse of food simulant E (PPPO) after repeated migration tests and several cycles of Soxhlet. A multilayer film composed of PET/Al/PE (food contact) was used as model material since common migrants such as alkanes, erucamide, dibutyl sebacate and lactones migrated from this film into PPPO. The homogeneity of the selected migrants in this film was assessed by the quantification of their initial concentration in the film after extractions using dichloromethane and by the values from the first migration assay. Most migrants were homogeneous in the film in at least one of the tests.

Performance characteristics such as linearity, precision, recovery and matrix effect (ME) of the analytical method for determining the selected migrants in PPPO were evaluated. The ME occurred for some substances but using the matrix-matched calibration curves this effect could be suppressed. The method showed good precision and recovery at the migrated level and it was considered efficient for quantification of the substances that migrated from the PET/Al/PE film into PPPO.

Changes in the particle size and shape of PPPO could not be clearly observed by optical microscopy. Consequently, the particle size was also assessed by sieve analysis. The results indicated that no major changes occurred in the particle size after performing 11 migration tests and 45 cycles of Soxhlet on the same PPPO portion. Although similar particle size distribution between the ranges of 180 and 250 µm (90%) was observed when compared to the virgin PPPO (95%), it was also possible to conclude that less amount of PPPO was retained in the sieve of 60 mesh (250 µm). An increase in the amount of PPPO in the sieves smaller than 180 µm was also observed.

The migration results indicated that PPPO should not be recycled extensively in the same cleaning protocol for different classes of substances. It was found that when 1 to 3 Soxhlet cycles were performed between two migration tests no pronounced decrease in the migration values was detected. It seemed that cleaning by Soxhlet exhibited a greater effect in the decreasing of migration values than the repeated migration tests including one solvent extraction step. A more noticeable decrease in the migration values could be observed when 5 and 10 cycles of Soxhlet were carried out between the migration tests, i.e., after 8 repetition migration assays and 20 cycles of Soxhlet. Taking into account the substances tested in this research and the results obtained, it can be stated that it may be feasible to recycle this food simulant up to 20 times by Soxhlet using acetone. This recycled PPPO could be reused in the migration assays if no more than 3 cycles of Soxhlet were performed between two migration tests.

However more studies should be done to clarify if the effect of the Soxhlet is more aggressive than the migration test itself. Additionally reference packaging materials and model chemical substances, to cover different polarity and functional groups, should be analysed in order to foresee either a guideline or a standard operation procedure for quality assessment for (recycled) food simulant E containing recommendations about the quantification of migrants using a matrix-matched calibration.

Note on output: A publication is in preparation for submission to a peer referred journal.

2.4 Coordination of EURL activities for the application of new analytical methods;

A new initiative to coordinate and increase the effectiveness of NRLs is to set up communities of competences for NRLs. This would palliate to the current unrealistic financial obligations to have every NRL to be an expert in everything, a clearly impossible task for such a wide ranging field of materials and expertise. An updated compilation was done in 2015 of methods 1) in development, 2) validated and 3) accredited 4) in use/object of monitoring by the NRLs of the network. The results are made available to NRLs directly on the CIRCABC website.

2.5 EURL collaboration with laboratories in third countries.

A brief exchange with the Thai Department of Science Service took place in September 2015 during a visit of a large delegation of the conference Food Contact Compliance to the JRC site. It is foreseen to have further exchanges in 2016.

The JRC was invited by Marco Zhong (Director Consumer Product Laboratory of Inspection & Quarantine Technology Centre) who is heading the National Reference Laboratory for Food Contact in Guangdong. The Symposium was organised by the expert Committee of Food Contact Materials of the China Food Industry Association (FCS; www.foodcontactscience.org). E. Hoekstra made a presentation on the update on EURL-NRL network with a focus on the development of methods and guidance for implementation of EU frameworks and future options. A portion of the presentation was dedicated to the JRC technical guidelines on compliance testing for plastic FCMs in

support to the Regulation (EU) NO 10/2011 on plastics. A significant demonstration of interest received and the JRC confirmed that the guidelines had in their implementation plan the intention to be translated into Chinese. There also were presentations on e.g. release of nanoparticles, risk assessment of non-intentionally added substances (NIAS), legal compliance of coated packaging, Chinese legislation for FCM and practice of bacteria control in paper and board. Contributions also included a presentation from a NRL (NRL-Portugal) and the forum included a representative of DG SANTE for the delegation to China. A separate forum aimed at controls and regulators took place to provide exchange on regulatory and risk management with a focus on compliance and migration. These technical discussions were of importance for a mutual understanding of EU/ China regulatory and compliance approaches. Exchanges will be maintained with Chinese counterparts as follow-up of the dedicated participation that took place in 2015. Other ad-hoc initiatives will be planned for the following years according to their relative priorities. The presentation of the JRC can be found in annex 1.

3. OPERATIONAL OBJECTIVE 2: Appropriate proficiency testing ensuring efficiency of control methods

The mandate of the Commission Implementation Decision the following aspects:

- *ensuring planning and initiation of comparative testing by EU-RLs in accordance with internationally accepted protocols;*
- *address underperforming related issues within the EU-RL network;*
- *coordination of EU-RLs, NRLs and the Member States for the planning of performance testing in the work programmes.*

3.1 Follow up to the proficiency test of metals from plastics in regulation 10/2011

Regulation (EU) No 10/2011 establishes new limits for metals migrating from plastics. A first PT was organised in 2014. It was testing for the first time the proficiency of official control laboratories and participants were free to use any analytical method of their choice. Based on the results of the PTs, a protocol for multianalyte methods for inorganics was produced. Since no methods are available from CEN or other sources as reference or internationally agreed, this is the first protocol that was developed on the basis of its performance in an ILC. The protocol was also given to the standardisation group ISO TC 166 in support of the business plan to include a method description for quantification and corresponding performance criteria. This works supports a future validation for this new mandatory testing for metals regarding specific migration from plastics. The method is included in annex 2.

3.2 ILC 2015_001: Optimisation of temperature control in migration testing protocols

The EURL as well as several NRLs have investigated the importance of the control of temperature on migration testing results. These studies indicated a relevant impact on migration results affecting several types of articles (in particular kitchenware). The difference in numerous samples was not due to inhomogeneity of the materials but rather due to differences in the exposure phase, where it was not feasible to maintain a constant correct temperature over the whole length of the test. Following these alarming findings that directly affect the decision making on compliance vs. non-compliance, a more stringent protocol needs to be developed, compared, validated and implemented. A

series of work items has therefore been developed for the next two years. In a first phase in 2015, a pilot exercise was organised for the NRLs and associated Official Controls Laboratories (OCLs) to carry out a migration test as they would normally do and to monitor the temperature of the simulant throughout the exposure.

Report:

Report of an inter-laboratory comparison from the European Union Reference Laboratory for Food Contact Materials. ILC01 2015 – Temperature control during migration tests by article filling.

Emmanouil Tsochatzis, Anja Mieth and Catherine Simoneau

Publication in progress

Abstract:

This report presents the results of an inter-laboratory comparison on the temperature control during migration tests by article filling organised by the EURL-FCM. Participants had to carry out a migration test by article filling of provided polypropylene cups (volume 0.3 L) with food simulant D1 (ethanol 50 %, v/v) for 2 h at 70°C to the best of their knowledge, monitor the temperature of the food simulant inside one of the test specimen during the exposure phase and provide details for the operating procedure. The participation to that ILC was satisfactory. The laboratory performance was judged as moderately satisfactory considering the breadth of variations in the operating protocols of the exercise and the different effect of many and different factors.

Based on the results a root-cause-analysis will be performed as to set up recommendations for migration tests by article filling. This should greatly improve the migration testing carried out at temperatures at or above 40 degrees, which constitute 90% of the tests performed in FCM migration testing.

3.3 ILC 2015_004: Cluster ILC to improve cost effectiveness

Regulation (EC) No 1935/2004 supports the food safety for FCMs. For plastics Regulation (EU) 10/2011 represents close to 900 chemicals. Yet, only 28 regulated substances employed for FCM have a CEN method, and candidate methods from petitioners are far from readily available.

This project aims to develop a cost effective approach for testing compliance. The work item consisted of a dedicated ILC on the general capabilities of laboratories to conduct a specific migration test that included several analytes.

Report:

Report of an inter-laboratory comparison from the European Union Reference Laboratory for Food Contact Materials: ILC02 2015 – Specific migration from a multilayer pouch in Simulant A

Emmanouil Tsochatzis and Catherine Simoneau

Publication in progress

Abstract

This report presents the results of an inter-laboratory comparison on the Specific migration in Simulant A organised by the EURL-FCM, Ispra (Italy). Participants had to carry out a migration test, by immersion with food simulant A (ethanol 10 %, v/v) for 10 d at 60°C for a plastic material, quantify the substances caprolactam (CAP) and 2,4-di-tert butyl phenol (2,4-DTBP) after the migration with Simulant A, and provide details for the analytical and sample extraction procedure. The participation to that ILC was satisfactory. There were 29 participants to whom samples were dispatched. Homogeneity and stability studies were conducted. The z-score values, for each substance per laboratory, were obtained by the assigned value calculated with the results reported by the participants (robust mean). The participation of the laboratories in this ILC was very satisfactory.

3.4 Follow up actions of NRLs underperformance (ILC metals)

In ILC 03 2014 on Elements from Plastic Food Contact Materials[4], an underperformance of 4 NRLs was observed. Three NRLs were underperforming on the determination of iron while performing adequately for all other metals. One NRL underperformed only of the quantification of Antimony while all other metals (7) had adequate z-scores. Another NRL underperformed only for Zinc but with also satisfactory z-scores for all other 7 metals.

An investigation was conducted on the potential root causes of the underperformances. Analyses of all results and exchanges of information demonstrated that the underperformances were very limited (1 out of 8 metals in all cases) and that they were caused by random contamination and not by systematic errors.

Another ILC is planned in 2016 which will include both migration and quantification and the metals in question will be included again in order to verify the improvements for these NRLs and the confirmation of performance for all already well performing NRLs.

4. OPERATIONAL OBJECTIVE 3: Availability of scientific and technical assistance

This objective contains the following activities.

- *queries to EU-RLs for policy making and enforcement;*
- *initiation of EU-RLs collaboration with EFSA and international organisations;*
- *organisation of meetings for scientific and technical assistance from the EU-RLs;*
- *guidance initiation regarding analytical methods;*
- *networking activities for appropriate assistance by the EU-RLs.*

4.1 NRL expert workshop on ceramics

The workshop aimed at briefing NRLs and Stakeholders on the technical implication of the revision of the legislation on ceramics. It provided a complete overview of progress and new or updated methods. The workshop took place on October 12 2015. The workshop included the following aspects:

[4] https://ec.europa.eu/jrc/en/publication/eur-scientific-and-technical-research-reports/report-two-inter-laboratory-comparisons-european-reference-laboratory-food-contact-materials

1) Presentation of the work done over the last year and testing regimes for ceramics

2) Investigation of repeat use (3 migrations vs. 1 migration),

3) Comparisons made between different testing reagents (tomato sauce (TS) at 70 °C and citric acid (CC) 2hrs with 70 °C) were compared to the benchmark test acetic acid (AA) 24hrs 20 °C

4) Tests on rim to compare the different protocols

5) Work on crystal ware, with comparison of white wine (WW) and acetic acid at 22 °C and exploratory kinetics of wine and acetic acid

5) Investigation of accelerated tests

6) Investigation on the impact on migration results when a week end interrupts the testing of repeated use

7) Preliminary experiment on bakeware.

A presentation from JRC was made on crystal glass, which compared kinetics between 4% AA and the real food i.e. WW for different lengths of times from 5 min up to 24 hours, and repeat testing (3 consecutive migrations). The results showed that different testing liquids AA and WW showed similar results, therefore AA could be considered as an adequate simulant and that for lead (Pb) the release decreased with the repeated migrations.

A presentation was also made by NRL Germany (BfR) on the release of Pb from crystalware, where it was noted that release of Pb into synthetic tap water could increase with repeat exposure, unlike acid and foods.

Work was presented by the Stazione Sperimentale del Vetro on occasional use for crystal ware. A survey was conducted in France with 1000 respondents.

Further results specific to ceramics were also presented by JRC. A comparison was conducted on the effect of a week end (gap) during the migration test. Successive migrations were conducted where one of the migrations was separated by a week end. The articles were left during the non testing time with water or without water (empty) in order to assess which protocol would have the least effect compared with conducting 3 consecutive migrations of 3 successive days without gaps. The results without water left in an article over the weekend when the item is not tested showed minor only difference to 3 consecutive testings. The comparisons were conducted over a range of products to increase the confidence in drawing general conclusions. The results with a gap over the weekend end and the articles left empty (rather than filled with water) were more similar to 3 consecutive testing as would be the normal practice.

Finally the JRC illustrated the preliminary results obtained on the first samples of bakeware. Real food (e.g. tomato sauce) was used to in real food contact (boiling). Comparisons were conducted with AA at 4% during 24hrs at 22°C. This conventional test was found to be a worse case than tomato sauce boiling during 6 hours. Samples to be provided by industry members from associations were discussed (types, shapes, volumes) in order to conduct more thorough studies. 5-6 representative samples were agreed at the end of the meeting (12 samples of each).

4.2 NRL expert forum on compliance for kitchenware

European legislation on FCMs is long standing and has become extensive for plastics. In 2014 several (technical) guidelines in the framework of the implementation of Regulation (EU) No 10/2011 for plastic FCMs were being published or finalised. In particular the JRC has been entrusted to develop the technical guidelines supporting migration testing under Regulation (EU) No 10/2011.

This work will be completed in 2016 and therefore a workshop needs to be organised to take stock of the gaps that are still present for kitchenware which are made of a multitude of materials and often not regulated at EU level. The plenary included a session to foresee the formation a dedicated task for a drafting in 2016 and to list points to consider for the update. The work item is on track for its next phases.

This work item aims to provide a most state of the art consensus advice to convey to competent authorities and stakeholders. This will anticipate safety by design for articles not only manufactured in the EU but provide a critical guidance for imported articles.

4.3 Plenary Workshop for NRLs

The workshop served to strengthen the structure of the network and to identify the latest development that can be useful for the work of the NRLs. Specific topics concerning the specific analysis of FCMs were addressed during the workshops and the agenda included a discussion of results of the ILC follow-ups and current ILCs, as well as a session of general exchange of information and information from the Commission.

Report:

Proceedings of the EURL-FCM training workshop 2015 "Science collaborations behind safety in innovation for FCM" 22 September 2015 Ispra

C. Simoneau (ed)

JRC98592, ISBN 978-92-79-53913-8 (PDF), doi:10.2788/44507 (online).

4.4 Provision of expertise to Commission, member states, NRLs and EFSA

The activity encompasses support by means of information and technical advice to National Reference Laboratories, Commission services and EFSA. The work also included maintaining close awareness of developments in methodologies, report and give advice, as relevant in Workshops and/or on an ad-hoc basis. In addition the EURL-FCM will also liaise via e-mail and via the CIRCABC platform to ensure rapid flow of information. Examples of activities include: providing support to DG SANTE in technical matters concerning analytical methodologies for FCMs, if requested, participation to DG SANTE WG and/or EFSA meetings or working groups where indicated. It also included research activities in support to commission and providing information and advice on the use and training opportunities of the FACET RTD tool for the exposure assessment of contaminants from FCMs.

The activity provided support to DG SANTE in technical matters concerning analytical methodologies for FCMs when requested, participating in DG SANTE Working Groups (WG) meetings or other Commission WGs where indicated, performing research activities in support to commission e.g. ceramics, recycling, guidance to Regulation (EU) 10/2011. It included participation to the EFSA Food Ingredient and Packaging (FIP) Unit network to present the work on Technical guidelines on compliance testing for plastic FCM. Finally it provided information and advice on the use and training opportunities of the FACET RTD tool for the exposure assessment of contaminants from FCMs. The deliverables can be seen in the section outputs.

The activity also provided support by means of information and technical advice to NRLs. The work included maintaining close awareness of developments in methodologies, report and give advice, as relevant, at the Annual Plenaries, Workshops and on an ad-hoc basis.

No requests for training of laboratory personnel from NRLs were received. The ad-hoc questions the EURL or JRC FCM staff received were as follows: about 40-50 emails were received regarding information on FCM legislation (±70%), analytical methods (±20%), availability of standards (±5%) etc. All were timely replied to and the high satisfaction level of the response recorded for quality purposes.

4.5 Networking activities and external presentations

01-2015: presentation to ISO TC 166 Ceramics

The meeting was the 1st meeting of ISO TC 166 following its recent revival in 2014. The meeting took place in Paris over 2 days. Delegations included: Belgium China, France, Ireland, Italy, Netherlands, United Kingdom, and the ISO secretariat (Japan chairing, France co-chairing). The aim was to create a new work item for the work of ISO on new standards by opening a resolution to form both the TC 166 and a dedicated working group to redraft updated versions of the standards ISO 6486-1[5]. It also intended a review of the ISO 7086-2[6] and ISO 8391[7]. The JRC was invited as permanent "A-liaison" for the European Commission (acting also on behalf of DG SANTE). In this first meeting C. Simoneau presented the results of the on-going JRC studies as background to develop new test methods for foreseen changing limits in the EU. The work of the JRC was considered supporting evidence and raison d'etre enabling revisions of the standards, in particular with the work on new validation data supporting the possibility of adding ICP MS an analytical technique of choice. In addition the extensive work covering ceramics and crystal ware gave the basis for updating the standards.

03-2015: 4th Fresenius Conference "residues of food contact materials in food"

The participants included Member States competent authorities, professional associations and representatives of DG SANTE (Jonathan Briggs), EFSA (Eric Barthelemy). The first session of the conference focused on the review EU legislation to FCM, to EFSA guidance for safety assessment, EFSA BPA risk assessment, Council of Europe work, use of bioassays, body burden from mineral oils. It provided specific updates on the technical guidelines for migration testing drafted by the JRC, as well as the work on ceramics in support to the revision of the Directive. The second session included presentations on pilot projects, migration from printing inks, migration of nano-additives, hydrocarbons and mineral oils from paper and board, efficacy of barriers for recycled paper and board, non-targeted analysis for food contact, risk and exposure to NIAS. E. Hoekstra gave an overview of the technical guidelines that were drafted by a special task force over the past 2 years led by JRC. The presentation also included show and tell with one typical test case scenario. C. Simoneau made a presentation on the work carried out by the JRC in 2014 on ceramics, enamel and crystal ware, as part of the EURL work. This was also followed by a session on questions and answers.

03-2015: Workshop on Agrifood collaborations EU China

A participation took place to a workshop on Agrifood collaborations EU China organised by DG AGRI. The overall policy framework on Research and innovation was presented by

[5] Ceramic ware, glass ceramic ware, and glass dinnerware in contact with foods- release of Cd part 1- test methods. Part 2 admissible limits
[6] Glass hollowware in contact with food- release of Pb and Cd,
[7] Ceramic cookware in contact with foods: release of Pb and Cd

DG RTD. As well as the policy dialogue on R&I in Food, Agriculture and Biotechnology – FAB Flagship by DG AGRI. DG SANTE presented the food standards in the EU and cooperation with China, including the revision of the Chinese Food Safety Law. In a second phase, projects and laboratory collaborations were presented. The JRC had a presentation on EU-China laboratory collaborations on chemical safety of materials and articles in contact with foods both as JRC and in the context of its EURL-FCM role. Other presentations included the FOODINTEGRITY FP7 project (Matthew Sharman, FERA, UK), the LinkTads FP7 project (Renata Clarke, FAO). The meeting was organized into 2 working sessions around the themes risk assessment and risk management. The work of the JRC enabled initiatives for collaboration for the field of food safety and FCM. During the meeting, several areas were identified (meat, dairy) and it was also pointed out that FCM is a standard cross over horizontal issue in food safety since packaging is inherent requirement of presence sold food and food preservation. The meeting was an interesting networking opportunity to promote the area of FCM to FAB initiatives organized towards EU-CN collaborations. The presentation of JRC to DG AGRI led to the development of a new call in the context of H2020 and Food, Agriculture, Biotechnology (FAB) Flagship initiatives (new call SFS-45-2016 with special focus on packaging).

07-2015: 2nd meeting of the scientific network for risk assessment of regulated food ingredients and food packaging "FIP Network"

The invited participation's purpose was to contribute and present relevant work in the 2nd meeting of EFSA's scientific network for risk assessment of regulated food ingredients and food packaging "FIP Network". The meeting composed of representative of Member State and risk assessment bodies focused on the work of the subgroup on FCMs dealing with risk assessment activities, approaches and challenges related to non-plastics; cooperation to support and to harmonise risk assessment practices. A portion of the meeting focused on the presentation and discussion of the EFSA CEF Panel draft opinion outlining the scientific rationale for revisiting the current guidance. Other documents, draft guidances and presentations followed. In particular E. Hoekstra presented the JRC Draft guidelines on migration testing in support to Regulation on food contact plastics Regulation (EU) No 10/2011. The Netherland (B. Van de Ven) presented a draft Guidance on NIAS. The Council of Europe CoE (S. Bahrke) presented an overview of their current activities. The European Commission /DG SANTE (G. Gallhoff) gave a short brief on the baseline study undertaken on their behalf by the JRC and currently in progress. The UK (R. Burden) presented the activities and approaches past and present. Belgium (F. Bolle) presented the new regulation on coating. Germany (S. Merkel) presented a research project on migration from elastomer for food contact". An update on MS Cooperation and outcome was given on a collaboration France-Denmark Research project on quantification and risk assessment of "unknown" contaminants migrating from Paper and Board. The meeting was concluded with some proposals for follow up in terms of scientific cooperation and future. In particular the work of JRC related to the baseline study was noted as relevant for the next meeting and updates. It was also noted with much interest the JRC involvement in a group formed by professional associations to draft technical guidance for testing materials that do not fall under the scope of the regulation on plastics.

09-2015: visit of a delegation from the conference "Food Contact Compliance"

In September, the FCM activities accommodated the largest group ever to the JRC premises on the Ispra site. The group was made up of the participants to the 3 day International Conference on Food Contact Compliance held in Baveno on the occasion of EXPO 2015. The organisers of the event dedicated an entire afternoon to visit the JRC facilities. We managed to welcome and organise for all of the 120 visitors with a guided tour of the Visitors' Centre, a presentation the meeting room and a bus tour of the site and refreshments with a group photo.

09-2015: Conference FCM Luxembourg " Conference: "Food contact materials: working together for safety and innovation in Europe"

The Ministry of Health in Luxembourg organised a Food Contact Materials conference on the 30th September in Luxembourg-Kirchberg as part of its Presidency of the Council of the European Union. The conference aimed to bring together different representatives working in the FCMs area such as the European Commission, the Council of Europe, the European Food Safety Authority (EFSA), Member States experts and representatives from the industry. The main focus lies on safety and innovation. How can the safety of FCMs be ensured in order to protect consumers? What kind of challenges is the area confronted with? What advantages does innovation bring to both consumer and industry? Attendees were selected from key stakeholders whose interaction aimed to create a cross-sectorial, highly relevant and dynamic discussion forum. These participants included decision makers, representatives from the European Commission, Members of the European Parliament, operator organisations, and European umbrella organisations representing interest groups and associations actively engaged in the field of FCMs. Each session included panel discussions as well as Q&A sessions to allow best possible involvement of all participants.

A first session focused on risk assessment. The session aimed to provide a forum on evaluation systems, as well as address the issue of missing analytical methods for a large number of substances. A forum then focused on how can the protection of the consumer be ensured? Which are the necessary steps? The chair of this session was Georges Kass (EFSA), and panel members also included Catherine Simoneau (JRC), Paul Hunt (expert for EuPIA) and Dr. Françoise Godts (expert for PlasticsEurope).

A second parallel workshop focused on Official controls which addressed the issues and challenges of official controls at different levels, e.g. inspection and laboratory legislation. The session was composed of chair Gudrun Gallhoff (DG SANTE) and Panelists Mette Holm/Carina Mørkhøj (Danish Veterinary and Food Administration), as well as Gregor McCombie (Food Safety Authority Zürich, Switzerland).

A third parallel workshop focused on innovation, i.e. new and future trends in the FCMs area. Which direction does the research take? How can the safety of new products be ensured? What advantages do these new products have?. It was chaired by Laurence Castle (ex- FERA) , with panelists Horst-Christian Langowski (TU Munich / Fraunhofer Institute for Process Engineering and Packaging), Regis Heyberger, (Plastipak Holding Inc.), Fabien Bolle (Belgium Scientific Institute of Public Health) and Lorenzo Zullo (European Tyre & Rubber Manufacturers Association).

11-2015 Invited presentation to the conference RAFA 2015

RAFA (Rapid Advances in Food Analysis) 2015 is an international conference related to the field of analytical chemistry applied to food safety and authenticity. Experts from academia, research centres, administration and industries were present and they exchanged on the latest scientific advances in this area. The main topics of the conference were recent issues and novel technologies, bioanalytical methods for food control, flavour and food quality markers, food allergens, food authenticity and fraud, food contaminants and residues, general food analysis, natural toxins analysis and omics approaches in food analysis. C. Simoneau presented the technical challenges and achievement of the EURL-FCM in a session dedicated to experience, challenges and achievement of selected EURLs in the field of food safety. The session was heavily attended with more than 170 people. The conference was an opportunity to disseminate the work, challenges and achievement specific to FCMs and to have an comparison to the challenges faced by the other EURLs in food chemical and microbiological safety. Being one of the 5 selected (out of ca. 28) was a positive indicator of the importance attributed to FCM in the overall food safety framework of EURLs. The session was considered a success by the chairs considering there were several parallel sessions on different topics.

11-2015/03/2016 Participation in the Council of Europe

The EDQM is the European Directorate for the Quality of Medicines and Healthcare. The meeting focused of the Committee of Experts on packaging Materials for food and pharmaceutical products). 8 countries were represented in the meeting. The presence of the JRC was to represent the technical link to Commission services is very relevant for the Council of Europe. This was underlined multiple times over the course of the 2-day meeting.

4.6 Reciprocal exchange of information with professional bodies and stakeholders

This activity is dedicated to provide support to standardisation bodies such as CEN and ISO for the standardisation of analytical methods for the determination of migrants from FCMs. The results of activities on FCM are updated on the JRC Science Hub under the link https://ec.europa.eu/jrc/en/research-topic/food-contact-materials and the site specific to the EURL https://ec.europa.eu/jrc/en/eurl/food-contact-materials. The JRC Science Hub platform supports the public dissemination of the work on food contact and serves as a reference, contact and service point for laboratories involved in the analysis of FCMs in Europe and worldwide. The website holds information about the activities and events carried out by the EURL-FCM as well as published reports available and scientific papers. The Network of NRLs is also emphasised.

A **functional mailbox** JRC-FCM@ec.europa.eu that serves as **help desk** for questions, was used about 50 times in 2015.

The dedicated website on CIRCABC specifically for NRLs is designed to support dissemination of information and repository of documents under the JRC EURL FCM (https://circabc.europa.eu/). The platform represents a main source of information exchange between the EURLs and the NRLs as well as is used for repository of working documents and traceability purposes. It also holds forms, sheets and other documents thus facilitating the management of tasks. It was updated together with the list of NRLs contacts. The activities also promoted invited participation/presentation of EURL activities in international conferences in the area.

Providing support to standardisation bodies such as CEN for the standardisation of analytical methods for the determination of migrants from FCMs. This requires a regular participation in the meetings of CEN TC 172/WG3 and ISO TC 166 where applicable.

5. 2015 SPECIFIC FCM PRIORITY 1: Pre-normative support to development of a regulation for ceramics

The Regulation on ceramic materials and articles is currently under revision. Under this revision a significant reduction of the limits for lead (Pb) and Cadmium (Cd) is foreseen. The current DG SANTE work programme with Member States highlights that a reduction of the existing limits implies the need to investigate changes in methodology, which has led the necessity of this work. The regulation of other metals through introduction of migration limits may need to be considered as well. In addition, crystal glass materials and articles may be brought into the scope of the regulation. The work programme includes a number of large deliverables related to the development and validation of new and improved methods for testing metals migration from ceramic materials in the context of the revision of Council Directive 84/500/EEC, as follows:

5.1 Migration method from ceramics towards new limits for selected metals

The PT of 2014 was used to 1) generate a full method protocol, which was also given to the standardisation group ISO TC166 WG3 on ceramics. 2) cluster data from different methods and derive precision criteria for each methods, which was also given to TC 166 WG3.

A presentation was done on January 26-27 2015 and is included in Annex 3.

Report:

Precision criteria of methods for the quantification of metals migrated from Food Contact Materials: Pre-validation data derived from ILCs on elements representative for plastics and ceramics

Giorgia Beldì, Natalia Jakubowska and Catherine Simoneau

Report EUR 27827 EN, JRC100838, ISBN 978-92-79-57670-6 (PDF), ISSN 1831-9424 (online), doi:10.2788/592775 Luxembourg: Publications Office of the European Union, 2016

Abstract:

Precision criteria from ILC03/04 2014 data, including repeatability and reproducibility standard deviations for the quantification of metals in acetic acid 3% and acetic acid 4% migration solutions for different analytical techniques were calculated and presented in this report. Three groups of analytical techniques were identified: Inductively coupled plasma mass spectrometry (ICP-MS), inductively coupled plasma Optical emission spectrometry (ICP-OES) and atomic absorption spectrometry (AAS). AAS includes graphite furnace (GF-AAS) and flame (F-AAS). Two laboratories provided results obtained using two different techniques. Laboratories which did not provide details of analytical technique used were not taken into account. Precision criteria were calculated applying robust statistic methods with algorithm A+S (ISO 5725-5 and ISO 13528)[2,3] and DIN 38402 A45 (Q-method/Hampel-estimator)[4]. The robust means calculated according to both algorithms were checked for significant difference (student's t-test) and equivalence test. Relative Repeatability standard deviations were very low generally less than 2% and reached 4.45% only for Pb in sample C2. Relative

reproducibility standard deviations for Pb were around 10% for ICP-MS and ICP-OES techniques raised till 16% when AAS technique was used. Relative reproducibility standard deviations for Cd were around 5%, 10% and 20% using respectively ICP-MS, AAS or ICP-OES techniques. For other elements the relative reproducibility standard deviations obtained were for most of the cases less than 10%. From comparison study it is possible underline that the analytical techniques are generally equivalent and not significantly different for all elements and both samples. Only for Fe in PL1 the ICP-MS and ICP-OES resulted to be significantly different.

Based on the results, a further ILC will be organised in 2016 at EU level to generate precision criteria on the migration test itself in addition to the method of quantification.

5.2 Method for testing migration of rim

Currently there is no specific provision stipulated in the EU legislation for rim. The intention is to include limits also for the rim, which implies the development of an internationally agreed test. Test methods were compared on series of different articles of varied nature. Based on the results, a further full validation at EU level may be planned in collaboration with ISO TC166 to generate a new reference methods.

Report:

Scoping investigations on the release of metals from the rim area of decorated articles In support of the revision of the Ceramics Directive 84/500/EEC, 2016

Mercedes Ana Peltzer, Giorgia Beldi, Natalia Jakubowska and Catherine Simoneau

Report EUR 27178 EN, JRC95336 ISBN 978-92-79-47228-2 (PDF), ISSN 1831-9424 (online), doi:10.2788/484454 Luxembourg: Publications Office of the European Union, 2015

Abstract:

In the present work, the release of metals from 14 samples (9 industrial samples, 2 ad-hoc manufactured ceramic cups and 3 ad-hoc manufactured glasses) was investigated in order to provide underpinning data on migration of metals under different conditions from different samples. The rim area was tested using 2 different approaches (wine and a wine-simulating acidic liquid, i.e 4% aqueous acetic acid). The test using the simulant acetic acid 4% (AA4%) was further investigated using two different protocols (with and without the use of paraffin wax). The release of metals from glass samples were also studied into white wine as benchmark food. All the samples tested in this study released Pb. It was also possible to detect limited release of Cd and other metals from the rim area of decorated articles. It was observed that the release of Pb, Cd and other metals generally decreased in successive migrations. Highly decorated articles with very bright colours led to a greater release of metals. The release of elements from test articles into white wine was always lower than that into acidic simulants. The testing using citric acid at higher temperature and shorter time was not significantly more severe than that using acetic acid 4% and depended on the metals considered and potential damage of the heterogeneous decoration of the articles during the exposure. Overglaze decorated samples were more sensitive to exposure to food simulants and released metals to a greater extent than test articles with underglaze decorations. The use of melted paraffin wax on the non-rim area may gave lower migration of metals into food simulants and could be more realistic, but might not be necessarily relevant when considering the standard deviation of the results.

5.3 Method for testing migration from glassware

Currently there is no specific provision stipulated in the EU legislation for domestic crystal/glassware. Testing methods were developed for crystal ware, which were compared to testing on food itself (drinks) in order to develop fit for purpose conventional tests that can be both worst case scenario, but in line with exposure and with a pragmatic protocol from a laboratory standpoint. Based on the results, a validation at EU level may be planned in collaboration with ISO TC166 to generate new reference methods.

Report:

Scoping investigations on the release of metals from crystalware in support of the revision of the Ceramics Directive 84/500/EEC, 2015

Mercedes Ana Peltzer, Giorgia Beldì, Natalia Jakubowska and Catherine Simoneau,

Report EUR 27180 EN JRC95337, EUR 27180 EN, ISBN 978-92-79-47230-5 (PDF), ISSN 1831-9424 (online), doi:10.2788/885263, Luxembourg: Publications Office of the European Union, 2015

Abstract:

In the present work, the release from 15 samples of crystalware was investigated with respect to migration of metals in different conditions from different samples. Three test regimes were compared. The first one, currently in use for ceramic, foresees the use of acetic acid 4% as simulant and the test conditions are 22°C for 24 hours. This procedure was repeated three times to better represent the repeated use regime. Two alternative approaches: 1) citric acid 0.5% for 2 hours at 70ºC, three consecutives migrations and 2) pre-conditioning of the ceramic with acetic acid 10% during 5 hours followed by a single migration for 24h at 22°C with acetic acid 4% were investigated to shorten the time of analysis. The release of metals was also studied into white wine as benchmark food. For all samples included in the study a relevant release of Pb was observed. Only few other metals were found in leaching solutions: Zn, Sb, Ba and Ag. Both simulants presented higher migration respect to the white wine as a benchmark food. In general the release of metals seemed to be higher with citric acid than with the acetic acid in the first migration. Migration values obtained with pre conditioning step were above the third migration obtained with the conventional test with acetic acid.

5.4 Protocol for migration testing from ceramics and bakeware

Development for testing conditions to use for cooking/baking, towards establishing enforceability of new limits (e.g. Pb, Cd) for ceramics were extended to include testing conditions for migration from cookware/bakeware. Based on the results, a validation at EU level may be planned in collaboration with ISO TC166 to generate a new reference method.

Report:

Scoping investigations on the release of metals from ceramic articles in support of the revision of the Ceramics Directive 84/500/EEC, 2016

Giorgia Beldì, Natalia Jakubowska, Mercedes Ana Peltzer, and Catherine Simoneau

Publication in progress

Abstract

In the present study, the release of metals from 63 ceramic samples (61 commercial and 2 manufactured ad-hoc) was investigated under different conditions. Three different methodologies were compared and optimised. The first one currently in use foresees the use of acetic acid 4% (AA4%) as a simulant liquid and the test conditions are 22°C for 24h. This procedure was repeated three times to better represent the repeated use regime. Two alternative methodologies: 1) citric acid 0.5% (CA0.5%) for 2 hours at 70°C, three consecutives migrations and 2) pre-conditioning of the ceramic with acetic acid 10% during 5 hours followed by a single migration for 24h at 22°C with acetic acid 4% were investigated. The release of metals was also studied into tomato sauce as benchmark food. In addition the feasibility of consecutive test was evaluated and the results of multiple migrations with weekend time gap in between migrations were carried out with the aim to evaluate the overall effect on the final result of the compliance test. To verify that repeated use regime can be representative also for occasionally use ceramic articles, a study of storage effect on metals release were performed. Articles were analysed, stored for different time periods from six months to one week and reanalysed to check the possible trend in metals release. The testing conditions for articles of category 3a intended to be heated in the course of preparation of food and drinks were also investigated. A number of metals were found in leaching solutions (e.g. Al, Fe, Zn, Co, Li, Ba, Mn, V, Pb, Cd, Sb, Ti, Cr, Ni, Cu, As). In general the metals release seemed to be higher with the CA0.5% than with the AA4% test conditions. Both simulants presented higher migration with respect to the tomato sauce. The aim of the pre-conditioning was to accelerate the leaching process, but it did not achieve values of migration similar to those of the third migration of the traditional method AA4%. Obtained results show also that release tests can be conducted with any significant difference not only in consecutive way, when the ceramic articles will be leave dry at room temperature. For occasionally use articles, the storage time usually does not affect considerably the metals release. For cooking ware lead values of the first migration in acetic acid 4% at 22°C for 24 hours are always higher respect to those obtained after 6 hours with hot tomato sauce. Lead content coming from the first contact with tomato sauce is not always covered by results when repeated use regime test with simulant is applied. However, the lead release into tomato sauce during third kinetic are lower than those values obtained during third migration in food simulant.

6. 2015 SPECIFIC FCM PRIORITY 2: Improved enforceability of Reg. (EU)10/2011

Regulation (EC) No 1935/2004 supports the food safety for FCMs. For plastics Regulation (EU) No 10/2011 represents > 900 chemicals. Yet, only 28 substances have a CEN method, and candidate methods from petitioners can only be found for less than 20% of substances. The work item aims to develop sources for calibrants, develop a database of analytical methods for FCM substances authorised under Regulation (EU) No 10/2011 and develop a web site freely searchable, providing a one stop portal for checking compliance of FCM[8]. The work is a continuation of activities over the past year.

The deliverable included a number of outputs.

6.1 Repository of reference monomers and additives for regulated substances

The work on the availability of calibrants stems from a collection of substances that had been acquired over the years (yearly item) but have now to be renewed to reflect the current market. Work completed in 2015 is summarised below.

[8] note: following the deletion of Art 11(2) we no longer need a method for the 459 substances for which no SML is specified in Annex I. (which frees time to concentrate on substances for which we do need a method).

- the collection of monomers and additives was checked and renewed for substances that could be obtained
- Work was completed to establish sources of analytical standards from commercial sources updated to end 2015
- For those not available, a collaboration with the industrial association of chemicals related to FCMs (e.g. CEFIC) was requested and welcomed. The state of the art of the collection now completed was provided to CEFIC and it is expected to receive the missing sources over 2016-2017.
- The collection of substances that had been in the legacy of the JRC from submission of petitioners since 1996 and no longer complied with quality requirements for substances was phased out.
- A new repository of current substances was created for monomers, additives and starting substances.

Table 1 of Annex I of amended Regulation (EU) No 10/2011 (up to the 5th amendment) contains 868 FCM substance numbers. These are mostly single substances but there are also some mixtures. In 2015 the work focused to check the availability of calibrants since some inconsistencies with the collection stemming from the past were observed. A root cause analysis was performed and highlighted that inconsistencies appeared to be mainly caused by the differences in overviews that were extracted from the DG SANTE database and the Table 1 of Annex I of amended Regulation (EU) No 10/2011. A summary table in this report gives an overview of the current situation.

At present, 84% of the substances that have a SML of non-detectable have a calibrant available. This percentage becomes 71% for substances with a value (other than non-detectable) for the SML and 72% for substances that have a group SML. Few substances (10) have both an SML and a group SML. They have for 80% of them a calibrant available. The critical step or limiting step for the work of official controls can be found in the presence of substances that have the generic SML. IN this case, a calibrant is available for only 59% of them.

In 2015 further effort was made to look for the commercial availability of calibrants. About 30 substances were identified, either as new or as replacement of calibrants of which the quality cannot be guaranteed. Results of the inventory have been presented to CEFIC, who agreed to collaborate to complete the availability of calibrants.

Table 1 the number of FCM substances in Table 1 of Annex I of amended Regulation (EU) No 10/2011 and the availability of their calibrants and analytical methods differentiated for the different types of specific migration limits.

	FCM substances	Calibrant	Method	Calibrant + method
Generic SML	435	255	53	35
SML=ND	31	26	21	18
SML	266	189	159	127
Group SML	146	105	50	43
SML + group SML	10	8	5	4
Total	868	567	278	219

Note: "Total" = "Generic SML" + "SML=ND" + "SML" + "Group SML" – "SML + group SML"

6.2 Inventory of descriptions of methods of analysis

The work on methods stems from a collection of methods collected over the years (yearly item) from old dossiers of the ex-scientific committee for food as well as from EFSA and that must now be reviewed critically, consolidated, updated to latest availability and have remaining gaps evaluated.

In 2015, the EURL work consisted in the preparation for accelerated collection of new methods for which method descriptions will be required under Commission Regulation (EU) No 10/2011.

- All the method descriptions from SCF, SANTE archives were retrieved in 2014. Those only available as printout were scanned and text-recognised,
- EFSA petitions were accessed, reviewed and the methods being retrieved in 2014-2015.
- Communication with EFSA was established to be able to flag missing methods,
- Collaboration with CEFIC was organised for the same purpose of filling the gaps.
- A new repository of methods was created.

Table 1 of Annex I of amended Regulation (EU) No 10/2011 (up to the 5th amendment) contains 868 FCM substance numbers. These are mostly single substances but there are also some mixtures. In 2015 the work focused to check the availability of methods as similar inconsistencies to that of the collection of substances were noted for methods. SANTE database and the Table 1 of Annex I of amended Regulation (EU) No 10/2011. For 68% of the substances that have a SML of non-detectable have an analytical method available. This is 60% for substances with a value for the SML and 34% for substances that have a group SML. Few substances (10) have both an SML and a group SML. They have for 50% an analytical method available. The critical step or limiting step for the work of official controls can be found in the presence of substances that have the generic SML. IN this case, an analytical method is available for only 12% of the substances that have the generic SML.

For 58% of the substances that have a SML of non-detectable have both a calibrant and an analytical method available. This percentage becomes 48% for substances with a value for the SML and 29% for substances that have a group SML. Few substances (10) have both an SML and a group SML. They have for 40% of them both a calibrant and an analytical method available. IN the case of substances that have only the generic SML, the availability of having both a calibrant and an analytical method is available for only 8% of the substances. These substances are therefore the most problematic to control or to ensure compliance of.

For some substances the analytical methods in the EFSA dossiers are confidential. This problem has been notified to EFSA and DG SANTE. In the meantime CEFIC may help to solve this issue and to provide missing analytical methods.

6.3 Web portal for methods and sources of calibrants

The current listing of database of information on suppliers of calibrants and for methods will be transferred into a web accessible portal. It will link systematically the database of substance characteristics and availability to the database of methods, to provide a one-stop portal. This portal will become on-line and searchable. This work is still under development and will be delivered in July 2016.

7 - General tasks, impact, preparedness

- The Annual report for DG SANTE and National Reference laboratories of the deliverables of the EURL-FCM for 2014- financial report and technical report were timely submitted
- The proposal workprogramme and associated budget for 2016 was submitted for the operation of the laboratory.
- The Quality System (QS) implemented since 2003 continued overseeing, controlling and reporting upon the activities, ensuring they were executed timely and to the expected standards of excellence. It also made sure that the budget was properly allocated. Evaluation sheets as feedback from NRLs and Official Laboratories were collected to the European Commission when requested, as well as questionnaires and other relevant documents for traceability purposes.

The EURL plays a central role in the implementation of FCM legislation and in the quality and reliability of food control measures according to Reg. 882/2004. It ensures access to methods and standards for all stakeholders for and provides the means to ensure compliance and enforcement in harmonised manner. It allows a common interpretation of the EU legislation towards better consumer protection. This fosters confidence in products on the EU market.

The work with the Network improves compliance performance and advice given at EU level. It serves to the preparation of international standards levelling the global trade. It also conciliates safety and innovation by identifying potential issues of new materials.

New methods improved compliance of the new food simulant E established in Regulation 10/2011: 1) a method was developed validated at EU level by the JRC, 2) proficiency testings were organised with remedial actions, ensuring that reliable compliance can occur at EU level, and better standards for industry.

The development of new tests by JRC serves as basis to DG SANTE for the development of new limits for ceramics, bakeware, domestic glassware, crystal tableware. Key collaborations with industries represented more than 4500 samples. The JRC investigated release under conditions of use and developed pragmatic testing protocols for controls and industries. Results showed that lower limits do not pose an analytical problem but the existing test and lower limits will pose trade issues for hand crafted products produced by SMEs. This has enabled a second phase and JRC development of new tests mimicking better real use.

The EURL should have trained personnel available for emergency situations occurring within the Union (if appropriate) and to provide scientific and technical assistance to the Commission (and to EFSA), for expert technical /enforceability opinion, improvement of exchange of information and briefing to MS CA on support from and to NRLs, responding to MS CA and third countries queries on interpretation of technical information where need arise, establishing/verifying quality criteria for analytical data towards exposure assessment (EFSA), provide technical guidance to the correct implementation of the EU legislation, and in cases where the MS challenge the results of analyses. In 2015, the staff foreseen ex-ante matched the achieved ex-post. Permanent Staff dedicated to food contact for DG SANTE included 3 permanent researchers, and 3 permanent technical staff. Additional staff included 3-4 contracts. The permanent staff has 10 to 17 years for expertise on food contact and able to provide ad hoc contribution to DG SANTE but are not eligible to be covered under the EURL financial rules. 2 permanent staff are working specifically under accreditation ISO 17025 for the qualification of the EURL and one EURL contractual staff also does so. The EURL also has a functional mailbox for all stakeholders with quality criteria established for promptness (15 days) and quality of replies (acknowledgement of the help by stakeholders).

The JRC established its activity of food contact in direct support to DG SANTE in 1995. It responds to any ad-hoc request of DG SANTE E2 in its field in its capacity of support to EU policies of long standing expertise. JRC attends DG SANTE FCM working group meetings with Member States. It provides contributions where needed on either planned or ad-hoc basis. It also contributes to data and tools for EFSA where indicated in absence of conflict with its distribution of competences prioritising the service to DG SANTE. Currently the JRC drafts two technical guidelines in support of the Regulation on plastics (EU) No 10/2011 (migration testing and migration modelling). It also operated in supporting work to the follow up of a road map for non-harmonised materials.

A final indicator is used for publications in peer-reviewed journals and invitations as speaker to scientific conference, or presentations or posters/papers presented at conferences. The expected ex-ante was 1 publication in peer review journal; 3 presentation or posters/papers at conferences and 1 invitation as speaker and/or chairperson. The ex-post exceeded the targets and are summarised below.

Peer refereed:

The Determination of Key Diffusion and Partition Parameters and Their Use in Migration Modelling of Benzophenone from Low Density Polyethylene (LDPE) into different Foodstuffs, Joaquim Maia, Ana Rodríguez-Bernaldo de Quirós, Raquel Sendón, José Manuel Cruz, Annika Seiler, Roland Franz, Catherine Simoneau, Laurence Castle · Malcolm Driffield, Peter Mercea, Peter Oldring, Valer Tosa, Perfecto Paseiro, Food Additives and Contaminants - Part A Chemistry, Analysis, Control, Exposure and Risk Assessment, February 2016

Determination of primary aromatic amines in cold water extract of coloured paper napkin samples by liquid chromatography-tandem mass spectrometry. Oguzhan Yavuz, Sandro Valzacchi, Eddo Hoekstra, Catherine Simoneau. Submitted to Food Additives and Contaminants - Part A Chemistry, Analysis, Control, Exposure and Risk Assessment

Recycling Tenax®: effect of several cycles of cleaning and its reuse in migration assays. Juliana S. Felix, Catherine Simoneau. to be submitted

Presentations at international conferences

Update on EURL-NRL network: development of methods and guidances for implementation of EU frameworks and future options, HOEKSTRA Eddo; The 3rd Food Contact Material Safety Seminar, 10-11/12/2015, Guangzhou, China

Safety of plastic food contact materials: the challenges of "unknowns" and so called non-intentionally added substances, SILVA FELIX Juliana, VALZACCHI Sandro, RIPOLLES VIDAL Cristina, CAVALLARI Federico, CONTINI Claudia, HOEKSTRA Eddo, RIZZO Andrea, GATTI Alberto, SIMONEAU Catherine, 2015, EurofoodChem XVIII, 13-16/10/2015, Madrid

Food Contact Materials- challenges and achievements, SIMONEAU Catherine; 7th International Symposium on Recent Advances in Food Analysis (RAFA 2015), 03-06/11/2015, Prague

Technical guidelines for compliance testing in the framework of the plastic FCM Regulation (EU) No 10/2011 HOEKSTRA Eddo; 2015 2nd meeting of the scientific network for risk assessment of regulated food ingredients and food packaging "FIP Network" Subgroup on food contact materials 01-02/07/2015 Parma EFSA

China laboratory collaborations on chemical/food safety (with focus on food contact materials) SIMONEAU Catherine; DG AGRI, 2015, EU experts' workshop on food safety in the context of research cooperation between the EU and China, 23/03/2015

Technical guidelines for migration testing in the framework of the plastic FCM Regulation (EU) No 10/2011, HOEKSTRA Eddo, the 4th International Fresenius Conference "Residues of Food Contact Materials in Food", 05-06/03/2015

Update on migration testing for ceramics and plastics and implications for food contact tableware/ kitchenware, SIMONEAU Catherine the 4th International Fresenius Conference "Residues of Food Contact Materials in Food", 05-06/03/2015

EU Legislation and Risk Assessment HOEKSTRA Eddo, Adhesives: how could they affect food packaging safety, 19/02/2015, Zaragoza ES.

January 2015 Briefing for standardisation by ISO TC 166 on JRC EURL work on ceramics and crystalware (in support to a revision of the Ceramic Directive 84/500/EEC) SIMONEAU Catherine; PELTZER Mercedes Ana; JAKUBOWSKA Natalia; BELDI Giorgia;

Publications

Guidance for the identification of polymers in multilayer films used in food contact materials: User guide of selected practices to determine the nature of layers, Anja Mieth, Eddo Hoekstra, Catherine Simoneau, Publication in progress

Report of an inter-laboratory comparison from the European Union Reference Laboratory for Food Contact Materials. ILC01 2015 – Temperature control during migration tests by article filling. Emmanouil Tsochatzis, Anja Mieth and Catherine Simoneau, Publication in progress

Report of an inter-laboratory comparison from the European Union Reference Laboratory for Food Contact Materials: ILC02 2015 – Specific migration from a multilayer into food simulant A, Emmanouil Tsochatzis, Anja Mieth and Catherine Simoneau, Publication in progress

Proceedings of the EURL-FCM training workshop 2015 "Science collaborations behind safety in innovation for FCM" 22 September 2015 Ispra, C. Simoneau (ed), JRC98592, ISBN 978-92-79-53913-8 (PDF), doi:10.2788/44507 (online).

Prevalidation data on the quantification of metals migrated from food contact materials: precision criteria from two ILCs on elements migrated from plastics and ceramics food contact materials. 2016, Giorgia Beldì, Natalia Jakubowska and Catherine Simoneau, Publication in progress

Scoping investigations on the release of metals from the rim area of decorated articles In support of the revision of the Ceramics Directive 84/500/EEC, 2016, Mercedes Ana Peltzer, Giorgia Beldi, Natalia Jakubowska and Catherine Simonea, Report EUR 27178 EN, JRC95336 ISBN 978-92-79-47228-2 (PDF), ISSN 1831-9424 (online), doi:10.2788/484454 Luxembourg: Publications Office of the European Union, 2015

Scoping investigations on the release of metals from crystalware in support of the revision of the Ceramics Directive 84/500/EEC, 2015 Mercedes Ana Peltzer, Giorgia Beldì, Natalia Jakubowska and Catherine Simoneau, Report EUR 27180 EN JRC95337, EUR 27180 EN, ISBN 978-92-79-47230-5 (PDF), ISSN 1831-9424 (online), doi:10.2788/885263, Luxembourg: Publications Office of the European Union, 2015

Scoping investigations on the release of metals from ceramic articles and bakeware in support of the revision of the Ceramics Directive 84/500/EEC, 2016, Giorgia Beldì, Natalia Jakubowska, Mercedes Ana Peltzer, and Catherine Simoneau, Publication in progress

Annual report 2014 of the EURL-FCM on activities carried out for the implementation of Regulation (EC) no 882/2004 (2015) SIMONEAU Catherine

Reactivity assessment of nanoparticles: A new approach to biological surface adsorption index approach (BSAI) (2015) OMANOVIC-MIKLICANIN Enisa; VALZACCHI Sandro;

RAMOS FERNANDES Andreia; OJEA JIMENEZ Isaac; SIMONEAU Catherine; GILLILAND Douglas; ROSSI Francois;

Practical guidelines on the application of migration modelling for the estimation of specific migration (2015), HOEKSTRA Eddo; BRANDSCH Rainer; DEQUATRE Claude; MERCEA Peter; MILANA Maria Rosaria; STOERMER Angela; TRIER Xenia; VITRAC Olivier; SCHAEFER Annette; SIMONEAU Catherine;

Mieth A., Simoneau C. (2015) Report of an inter-laboratory comparison from the European Union Reference Laboratory for Food Contact Materials: ILC 02 2014 – Identifying the composition of multilayer plastic packaging films. Publication Office of the European Union, Luxembourg, JRC Scientific and Technical Research Report, EUR 27175 EN

Beldi G., Peltzer M.A., Simoneau C. (2015) Report of two Inter-laboratory comparisons from the European Reference Laboratory for Food Contact Materials - Elements from Food Contact Materials: ILC 03 2014 – Plastics and ILC 04 2014 – Ceramics. Publication Office of the European Union, Luxembourg, JRC Scientific and Technical Research Report, EUR 27167 EN

Silva Felix J., Beldi G., Simoneau C. (2015) Follow-up ILC01-2013 on Proficiency Testing on Food Simulant E containing a cocktail of potential migrant substances: Report of an Inter-laboratory Comparison from the European Union Reference Laboratory for Food Contact Materials: ILC01-2014. Publication Office of the European Union, Luxembourg, JRC Scientific and Technical Research Report, EUR 27166 EN

Annex 1- presentation to 3rd FCM Safety Seminar, China.

Annex 2 – method for the determination of metals under Regulation (EU) 10/2011.

Determination of Elements released from food contact plastic materials and articles into acetic acid 3% (v/v) using ICP-MS

JRC.I.1.WOP.FIT-EURL.004

SCOPE AND FIELD OF APPLICATION

The European Regulation (EU) No 10/2011 [1] in Annex II reports that "Plastic materials and articles shall not release the following substances in quantities exceeding the specific migration limits below:

Barium = 1 mg/kg food or food simulant.
Cobalt = 0,05 mg/kg food or food simulant.
Copper = 5 mg/kg food or food simulant.
Iron = 48 mg/kg food or food simulant.
Lithium = 0,6 mg/kg food or food simulant.
Manganese = 0,6 mg/kg food or food simulant.
Zinc = 25 mg/kg food or food simulant."

This procedure describes an ICP-MS method for the determination of Ba, Co, Cu, Fe, Li, Mn, Zn and also other elements such as Pb, Cd, Ag, Al, As, Cr, Mo, Ni, Sb, Sn, Sr, Ti, V and Zr in acetic acid 3 % (v/v) migration solutions obtained after the extraction test.

NORMATIVE REFERENCES

COMMISSION REGULATION (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food

ISO 17294-1:2005, Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) for the determination of elements — Part 1: General guidelines and basic principles

ISO 17294-2005, Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) for the determination of elements — Part 2: Determination of 62 elements

ISO 3696:1987, Water for analytical laboratory use - Specification and test methods

TERMS AND DEFINITIONS

ICP-MS	Inductively coupled plasma mass spectrometry
ANALYTICAL BLANK	Value determined by a blank sample covering the complete analytical procedure including extraction, clean-up.
BLANK CALIBRATION SOLUTION	Solution prepared in the same way as the calibration solution, but leaving out the analyte
CALIBRATION SOLUTION	Solution used to calibrate the instrument, prepared from stock solutions or from a certified standard
CHECK CALIBRATION SOLUTION	Solution of known composition within the range of the calibration solutions
INSTRUMENT DETECTION LIMIT	Smallest concentration that can be detected with a defined statistical probability using a contaminant-free instrument and blank calibration solution.
DETECTION LIMIT	The limit of detection is expressed as the mean analytical blank value plus three times the standard deviation of the analytical blank
QUANTIFICATION LIMIT	Limit above which a quantification of the measurands is possible, expressed as the mean analytical blank value plus, either, five to ten times the standard deviation of the analytical blank
WOP	Working Operating Procedure

PRINCIPLE

Determination of elements by inductively coupled plasma mass spectrometry (ICP-MS) consists of the following steps:

1. Introduction of a measuring solution into radiofrequency plasma to cause dissolution, atomization and ionization of elements;
2. Extraction of the ions from plasma through a differentially pumped vacuum interface and separation on the basis of their mass-to-charge ratio by a mass spectrometer;
3. Transmission of the ions through the mass separation unit and detection, usually by a continuous dynode electron multiplier assembly, and ion information processing by a data handling system;
4. Quantitative determination after calibration with suitable calibration solutions.

The relationship between signal intensity and mass concentration is usually a linear one over at least five orders of magnitude. For more details refer to ISO 17294-1:2005.

INTERFERENCES

It is important underline that when using ICP-MS, the presence of concomitant elements in the sample can cause interferences, for instance systematic errors in the measurement of the signal. Interferences are classified into spectral and non-spectral interferences.
The components that can cause spectral interferences are the following:

1. An isotope of another element having the same nominal mass-to charge-ratio as the analyte isotope, for example ^{58}Ni (analyte) and ^{58}Fe (interferant). Isobaric interferences may be corrected using the abundance of a different isotope of the interfering element. However

correction options are often included in the instrument software. The isotope for measurements can usually be chosen free from isobaric interferences.

2. Polyatomic or molecular and doubly charged ion interferences. In many cases this ions contain argon (plasma gas) and/or oxygen originating from the water of the solution aspirated, for example ^{56}ArO(interferant) and ^{56}Fe (analyte).

3. Doubly charged ions for instance Ba^{2+} interference with ^{65}Cu, ^{66}Zn, ^{67}Zn and ^{68}Zn.

Significant molecular and doubly charged interferences shall be corrected using elemental equations (e.g. ^{58}Ni = - 0.04825 ^{54}Fe), a collision or a reaction cell or other possible elimination strategies given in ISO 17294-1 paragraph 6.

Non spectral physical interferences are associated with the sample nebulisation and transport processes as well as with ion-transmission efficiencies. Nebulisation and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause relevant signal suppression or enchantement. Dissolved solids can deposit on the nebuliser tip of a pneumatic nebuliser and on the interface skimmers. Total solid levels below 0.2% (2.000 mg/l) are recommended to minimise solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the measurement element so that the two elements are similarly affected by matrix changes. Dilution of the sample fivefold will usually eliminate the problem. Detailed information on spectral and non-spectral interferences are given in ISO 17294-1 paragraph 6.

REAGENTS AND MATERIALS

6.1 Reagents

All reagents shall be of recognised analytical grade.

6.1.1 Distilled water or water of equivalent purity (grade 3 water complying with the requirements of ISO 3696) shall be used throughout.

6.1.2 Acetic acid, (CH_3COOH), glacial, ρ = 1.05 g/ml, CAS 64-19-7.

6.1.3 Acetic acid test solution, 3 % (v/v) solution
Add 30 ml of acetic acid (6.1.2) to distilled water (6.1.1) and fill to 1 l. This solution shall be freshly prepared for use. Proportionately greater quantities may be prepared.

6.1.4 Nitric acid, r(HNO3) = 1.4 g/ml.

NOTE: Nitric acid is available both as:
$r(HNO_3)$ = 1.40 g/ml equivalent to $w(HNO_3)$ = 650 g/kg;
$r(HNO_3)$ = 1.42 g/ml equivalent to $w(HNO_3)$ = 690 g/kg.
Both are suitable for use in this method.

6.1.5 Elements stock solution

Single-element stock solutions and multi-element stock solutions with adequate specification stating the acid used and the preparation technique are commercially available. For example element stock solutions with concentrations of the analytes of 1000 mg/l are suitable. These solutions are

considered to be stable for more than one year, but in reference to guaranteed stability, the recommendations of the manufacturer should be considered.

6.1.6 Standard solutions

ρ (Ag, Al, As, Ba, Cd, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Pb, Sb, Sn, Sr, Ti, V, Zn and Zr) = 10 mg/l
Pipette 10 ml of elements stock solutions of 1000 mg/l (6.1.5) separately or together, if suitable, into a 1000 ml glass volumetric flask. Add 10 ml of nitric acid (6.1.4). Bring to volume with water (6.1.1) and transfer to a suitable storage bottle.
Elements standard solutions are considered to be stable for several months, if stored in the dark. This does not apply to elements and multi-element standard solutions that are prone to hydrolysis, in particular solutions of Mo, Sn, Sb and Zr. In reference to guaranteed stability of all standard solutions, see the recommendations of the manufacturer.

6.1.7 Intermediate standard solutions

ρ (Ag, Al, As, Ba, Cd, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Pb, Sb, Sn, Sr, Ti, V, Zn and Zr) = 1 mg/l
Pipette 10 ml of elements standard solutions of 10 mg/l (6.1.6) separately or together, if suitable, into a 100 ml glass volumetric flask. Bring to volume with water (6.1.1) and transfer to a suitable bottle. Prepare the intermediate standard solutions freshly before each use.

6.1.8 Internal standard solution (reference element solution)

The choice of elements for the reference-element solution depends on the analytical problem. Solutions of these elements should cover the mass range of interest. Generally an internal standard should be no more than 50 amu removed from the analyte. The concentrations of these elements in the sample should be negligibly low.
The elements for example ^{45}Sc, ^{85}Y, ^{103}Rh, ^{165}Ho and ^{187}Re can be suitable for this purpose.
Internal standard solution, which contain one or more of proposed elements may be used: ρ (Sc, Y, Rh, Ho and Re) = 5 mg/l.
Pipette 5 ml of each element stock solution (1000 mg/l of each Sc, Y, Rh, Ho and/or Re) into a 1000 ml volumetric flask. Add 10 ml of nitric acid (6.1.4). Bring to volume with water and transfer to a suitable storage bottle.
A suitable concentration range of the internal standard in samples and calibration solutions is 10 - 100 µg/l.

6.1.9 Calibration solutions

Prepare the calibration solution(s) that cover the required working range by diluting the element standard solutions (6.1.6) or intermediate element standard solutions (6.1.7). Add an adequate volume of acetic acid 3% (v/v) (6.1.3) to make the composition of the calibration solutions equal to the composition of the test sample solutions to minimise the matrix effect. If necessary, add internal standard solution (6.1.8) to a concentration of for example 10 µg/l or 100 µg/l of the reference elements before bringing up to volume.

6.1.10 Calibration blank solution

The calibration blank solution is prepared in the same way as the calibration solutions, but leaving out the analytes. Prepare the calibration blank solution by adding an adequate volume of acetic acid 3% (v/v) (6.1.3) to make the composition of the calibration blank solutions equal to the composition

of the test sample. If necessary, add internal standard solution (6.1.8) to a concentration of for example 10 µg/l or 100 µg/l of the reference elements before bringing up to volume.

6.1.11 Initial calibration verification solution

The calibration verification solution is prepared by combining elements of interest from a standard source different from that of the calibration standard, and at concentration near the midpoint of the calibration curve. This standard may also be purchased. The solution should be prepared in the same acid composition (matrix) of the calibrations and the test samples.

6.1.12 Continuing calibration verification solution

The continuing calibration verification standard solution should be prepared combining metals of interest from the same standards used for calibration, at a concentration near the mid-point of the calibration curve. The solution should be prepared in the same acid composition (matrix) of the calibrations and the test samples.

6.1.13 Interference check solution

The interference check solution (ICS) is prepared to contain known concentrations of interfering elements (see paragraph 6 of ISO 17294-1) that will demonstrate the magnitude of interferences and provide an adequate test of any corrections, for example Molybdenum serves (if not presented in the samples) to indicate oxide effects on cadmium isotopes. The other components are present to evaluate the ability of the measurement system to correct for various molecular-ion isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system within quality control limits. These solutions must be prepared from ultrapure reagents or they can be obtained commercially.

6.1.14 Optimisation solution

The optimisation solution, commercially available, serves for mass calibration and for optimization of the ICP-MS apparatus conditions, for example adjustment of maximal sensitivity with respect to minimal oxide formation rate and minimal formation of doubly charged ions. It should contain elements covering the entire mass range, as well as elements prone to a high oxide formation rate or to the formation of doubly charged ions.

APPARATUS

7.1 Inductively coupled plasma mass spectrometer

The ICP-MS system includes:

- Sample introduction system (pump, nebuliser, spray chamber);
- Inductively coupled plasma (radio-frequency generator, load coil, torch);
- Quadrupole or time-of-flight mass spectrometer, capable of scanning a mass range from 5 m/z (amu)
  to 240 m/z (AMU) with a resolution of at least 1 m/z peak width at 5 % of peak height, or sector field mass spectrometer;
- Collision/reaction cell that can be pressurized with helium and kinetic energy discrimination for polyatomic interference attenuation;
- Process control and data processing equipment;
- Argon gas supply - high purity grade, i.e. > 99.99 %;

- Hellium for collision cell – Ultra high purity grade, i.e > 99.999 %;
- Optional autosampler or additional (peristaltic) pump.

For more detailed information on the instrumentation, refer to ISO 17294-1:2005.

7.2 Accessories

The stability of test samples and calibration solutions depends to a high degree on the container material. The material shall be checked according to the specific purpose. For the determination of metals in acetic acid 3% (v/v) leachates, high density polyethene (HDPE) or polytetrafluoroethene (PTFE) containers (e.g. falcon tubes and storage bottles) are allowed.
Immediately before use, all glassware used to prepare stock and standard solutions should be washed thoroughly with warm diluted nitric acid e.g. w(HNO_3) = 10 %, and then rinsed several times with water (6.1.1).
The use of piston pipettes is permitted and also enables the preparation of lower volumes of calibration solutions. The application of dilutors is also allowed. For more detailed information on the instrumentation, refer to ISO 17294-1:2005.

PROCEDURE

8.1 Instrument set up

Adjust the instrumental parameters of the ICP-MS system in accordance with the manufacturer's manual.
Wait at least 30 min to stabilise the plasma and adjust the instrument to working condition.
For guidance consult ISO 17294-1. For the selection of suitable isotopes refer to paragraph 6 of ISO 17294-1.
Use the recommended optimisation solution (6.1.14) to optimise or check the sensitivity and the stability of the system. Check the resolution and the mass calibration as often as required by the manufacturer. Define the relative atomic masses and the corresponding corrections. Define take-up and rinsing times to avoid memory effects.
Both standard mode or spectrum helium mode (KED - kinetic energy discrimination) are acceptable. KED mode should be used for elements with more interferences.

The use of an internal standard is recommended. Add the internal standard solution (6.1.8) to the interference check solution (6.1.13), to calibration solutions (6.1.9), to the blank calibration solutions (6.1.10) and to all test portions of samples before the analysis or add the internal standard solution (6.1.8) on-line using two channel sample-introduction pump. The mass concentration of the reference elements shall be the same in all solutions.

8.2 LOD and LOQ determination

LOD can be calculated from the standard deviation of the blank. It is expressed as the mean analytical blank value (x_{bl}) plus three times the standard deviation of the analytical blank (sd_{bl}).
For the calculation of LOD, 10 determinations of the blank samples are analysed according to the same analytical method and their standard deviation is calculated. The LOD of the whole method is calculated as: $LOD = x_{bl} + 3\ sd_{bl}$, where x_{bl} is the means concentration calculates from the counts of the noise peak for the 10 determinations; sd_{bl} is the standard deviation of the analysis.
LOQ, the limit above which a quantification of the elements is possible, is expressed as the mean analytical blank value plus, either, five to ten times the standard deviation of the analytical blank.

$$LOQ = x_{bl} + F\ sd_{bl}$$

The factor F depends to the accepted measurement uncertainty.

8.3 Calibration

When the analytical system is first evaluated, establish a calibration curve for the elements of interest using at least five measuring points (for example, the blank calibration solution (6.1.10) and four calibration solutions (6.1.9) over a linear range. The calibration range should encompass the elements concentrations of the sample.
The working range in general may cover the range of 0.2 µg/l to 200 µg/l or a part of this.
For work on a daily basis, one blank solution (6.1.10) and one to two calibration solutions (6.1.9) are enough to set up a calibration graph, but check the validity of the calibration curve with a certified reference sample, a standard sample, or a suitable internal control sample.
For more details refer to ISO 17294-1 paragraph 9.
Linear regression correlation coefficient (r) must be ≥ 0.998. If correlation coefficient is < 0.998, repeat calibration.

8.4 Determination of Ag, Al, As, Ba, Cd, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Pb, Sb, Sn, Sr, Ti, V, Zn and Zr

After establishing the calibration curves, measure the blanks and the interference check solution to establish interference correction or to check presence of interferences. Run the test samples and if the metals concentrations of the extraction solutions are found to be higher than the highest calibration point, dilute suitable aliquot portions to reduce concentrations within the working range with test solutions (6.1.3) or water (6.1.1) to have the same acidity composition of the calibration curve.
Within sufficient small intervals (for example, every 25 samples or less and at the beginning and end of the sample run) check the accuracy of at least one certified reference sample or one standard sample or a suitable internal control sample. If necessary, re-calibrate.

EXPRESSION OF RESULTS

State as many significant figures as are acceptable according to the precision of the measuring values, but not more than three significant figures.

QUALITY CONTROL

10.1 Blank

Result of the calibration blank check shall be within 3 times the instrumental detection limit.

10.2 Calibration verification and drift

Result of the initial and continuing calibration verification solutions shall not deviate more than 10%.

10.3 Internal standard abundance

Internal standard shell not deviate more than 20%.

10.4 Interference

The impact on the measured value of uncorrected isobaric, molecular and doubly charged interferences shall not be higher than 5% or three times the instrumental detection limit. Successive values of a correction factor shall not differ more than 20%.

PRECISION

An Interlaboratory trial, carried out among European reference and official control laboratories for food contact materials in 2014, yielded the results given in the Table 1. The sample used in the Interlaboratory trial was an acetic acid 3% (v/v) solution spiked with the elements reported in Table 2.

Table 1. Precision data for the determination of Ba, Co, Cu, Fe, Mn, Zn, Li and Sb in acetic acid 3% (v/v) using ICP-MS

Element	*l*	*n*	$\bar{x}$ [µg/l]	σ_R [µg/l]	Reproducibility CV [%]	σ_r [µg/l]	Repeatability CV [%]
Ba	26	100	693.31	31.90	4.60	10.09	1.46
Co	28	108	50.91	2.86	5.61	0.87	1.71
Cu	27	102	3062.18	146.77	4.79	36.04	1.18
Fe	25	94	19151.54	1675.18	8.75	337.13	1.76
Mn	26	98	494.65	24.50	4.95	7.42	1.50
Zn	24	90	20127.54	906.02	4.50	245.08	1.22
Li	22	86	505.01	26.92	5.33	9.80	1.94
Sb	25	100	34.49	3.92	11.38	0.57	1.65
l	is the number of laboratories;						
n	is the number of values;						
$\bar{x}$	is the robust mean;						
σ_R	is the reproducibility standard deviation;						
CV	is the coefficient of variation;						
σ_r	is the repeatability standard deviation;						
Ba, Co, Cu, Fe, Mn, Zn, Li and Sb were measured in acetic acid 3% (v/v) solutions. All precision criteria were derived applying robust statics without elimination of outliers (ISO 5725-5)							

Table 2. Sample used for the Interlaboratory trial

Sample matrix	Spiked element	*Concentration [µg/l]
Acetic acid 3% v/v	Ba	693.31
	Co	50.91
	Cu	3062.18
	Fe	19151.54
	Mn	494.65
	Zn	20127.54
	Li	505.01
	Sb	34.49
* robust mean from participants results		

SAFETY

General safety instructions should be followed at all times. All appropriate protective safety equipment should be worn and a fume cupboard must be used.

Annex 3 - performance indicators

The quality processes governing the functioning and evaluation of performance of the EURLs in food safety involve the setting of performance indicators that are set at the inception of the development of the work programme (ex-ante). Upon completion of the yearly work programme, these indicators are reviewed to assess that the body of deliverables are satisfactory both in terms of breadth and achievements.

FF.PT.1 Number of comparative tests following internationally standardized criteria ("high quality comparative tests") to be organised*	Expected ex-ante: 1) follow up to PT identification and quantification of metals from ceramics 2) ILC on temperature controls 3) ILC general migration testing capabilities (Specific migration). Achieved ex-post: 1) completed: harmonised protocol achieved 2) completed 3) completed
**If applicable, also expected number of comparative tests which are not performed following internationally standardized criteria, with information on number of test materials and analytes/parameters*	

FF.PT.2 Grading addressing the complexity of each PT with corresponding justification 1 - easy matrix, solution with analyte, one single easy analyte (relative high concentration) whose identity is shared with the participants 2 - more challenging analytes, concentrations and matrices 3 - more complex combination of analytes, concentrations and matrices whose identity is not disclosed to the participants, innovative substances, a mix of analytes either in the same matrix or in different matrices	Expected ex-ante: ILC-01: **3:** first evaluation of precision criteria of its kind for both plastics and ceramics on a range of >5 metals and by at least one technique. ILC 02: first attempts to identify and quantify the variations of temperature controls in migration testing ILC 03: first attempt to devise a specific migration testing including migration for article filling. Achieved ex-post: ILC01: **level 3:** Full precision criteria derived based on 8 metals for plastics and 8 metals for ceramics, in release values in the range of the new intended values (taking into account both DG SANTE and Council of Europe where applicable), and investigation the respective precision criteria of 3 different analytical instrumentation (ICP MS, OIES, and AAS). ILC02: **level 3**: never attempted before- no data exists, interpretation a new concept as semi qualitative data treated by cluster analysis and principal component analysis to derive interrelation between performance of temperature controls and practices ILC 03: **level 2**: several migrations , migration tests in addition to quantification, multilayer that involved good practice for testing
FF.PT.3 Average rates of NRL success (share of NRLs that are expected to meet all the test thresholds) in relation to PT's grade of complexity and methods and activities to ensure follow-up of poor results*	Expected ex-ante: ILC-01: target 70% satisfactory for metals under scrutiny for ceramics; ILC-02: not yet applicable; there are NO conventional /standardised/ validated methods; this exercise aims to assess the relevance of the issue. ILC-03: not applicable, never attempted before and does not exist in any ILC scheme worldwide so no benchmarking possible. Achieved ex-post: ILC-01: 92% Achieved beyond planned for participation (56 labs) and success (90%). ILC-02: 29% of the laboratories achieved full score. This highlights the outmost importance of this aspect that had never been assessed quantitatively before as an issue for official controls. This ILC is the most relevant starting point for work that had been identified by NRL as problematic and necessary for harmonisation ILC-03: 82-84% was satisfactory, which indicate a situation fairly acceptable as an exercise never attempted before.
**Follow-up work to significantly improve the performance of laboratories with poor results*	

FF.PT.4 Progress* (direct after training or based on past few years' experience) made by NRLs on similar comparative tests with possible discussion of influential factors	Expected ex-ante: 2015 ILCs: not applicable since first time ever exercises. Achieved ex-post: n/a yet
**Progress is understood as either a reduction of deviation (e.g. 5% instead of 10%) or a reduction in the number of NRLs failing a similar comparative test*	
FF.PT.5 Availability for use by NRLs of analytical methods (i) collected by the EURL* , or (ii) prescribed for official control by European legislation (if applicable)	Expected ex-ante: 1) databank containing methodologies. Target was 70% for substances and 60% for methods. 2) guidance for the analysis and identification of polymers Achieved ex-post: Calibrants – target achieved 84% of the substances with SML of non-detectable have a calibrant available. 71% for substances with a value (other than non-detectable) for the SML The issue of regulating substances using a generic SML is being addressed since this was uncovered as the main issue for enforcement, Methods- target achieved 68% of the substances that have a SML of non-detectable have an analytical method available. 60% for substances with a value for the SML have a method. The critical step or limiting step for the work of official controls can be found in the presence of substances that have the generic SML. 2) guidance for polymers was published/ target achieved.
**In areas such as FCM with no standards prescribed, with an approach allowing a wide range of modifications depending also on the available technical equipment and limited standardization, description of support of NRLs with analytical details (if applicable) - (ii) not applicable*	
FF.PT.6 Reported use by NRLs in relation to EURL's specific promotional activities* of analytical methods (i) guidances developed or published by the EURL (expected take-up), or (ii) prescribed for official control by European legislation (if applicable)	Expected ex-ante: guidance for kitchenware guidelines 80% method for Tenax® dry simulant 80% guidance for the analysis and identification of polymers derived from ILC - 80% (measurement of use available in 2017 at t+1 year of publication) Achieved ex-post: To be measured in 2016

FF.ANA.1 Number of newly available analytical methods disseminated to NRLs: description of the situation* with specification e.g. of new analytical methods developed by the EURL or in general, or description whether partially modified methods (with improvement in some steps) or completely new methods are expected	Expected ex-ante: The target is for the new substances that have been recently included in the latest amendment of Reg 10/2011 (e.g. Reg. 202/2014, 2015 if applicable). In the context of ceramics, two new methods will be developed for the new intended levels for ca 5 -6 metals including precision criteria. Achieved ex-post: New methods were identified for the substances included in the 5th amendment of the Regulation (EU) No 10/2011 as per the target set. Some of them are confidential in the EFSA dossiers which will require a further action on EFSA's part in coordination with DG SANTE.
**With regard to establishment of methods for FCM (e.g. methods obtained by EURL from the authorisation of new substances following an EFSA opinion, or from the re-evaluation of existing substances - if applicable; or from EURL development, or other projects providing advances in analytical methods specific to FCM).*	

FF.ANA.2 Number of calibrants retraced from analytical sources to provide the relevant information to NRLs or obtained by the EURL for the correct development and implementation of methods*	Expected ex-ante: To establish sources of analytical standards required as calibrants for the enforcement of Regulation (EU) No 10/2011.The current bank holds ca. 490 substances. Note: As there are no monitoring plans for FCM, it is not possible to establish what substances or to what extent substances may be needed or used to calibrate monitoring of control samples. the latest amendments will be given priority (ca. 10-20 substances) Achieved ex-post: Traced about 30 substances either new or replacing old calibrants for quality purposes.

FF.NRL.1 Number of participating NRLs in the annual workshop (attendance rate)/ Actions taken to ensure all NRL's participation	Expected ex-ante: 70%. Actions include several provisions: the dates are set in advance (normally at the biyearly plenary for the following year); Achieved ex-post: Plenary (annual workshops, n=2): June: Ex ante: 70% - ex Post (>75% attendance, calculation in progress) September: Ex ante: 70% - ex Post (>75% attendance, calculation in progress).
FF.NRL.2 Number of positive satisfaction surveys above 85% received for the annual workshop	Expected ex-ante: it is expected that more than 90% received survey will be positive surveys Achieved ex-post: June: Ex ante: 90% - ex Post 90% October: Ex ante: 70% - ex Post 90% (included in set of workshops)
FF.NRL.3 Measures to address relevant negative feedback from satisfaction surveys	Expected ex-ante: Specific questionnaire and periodical polls for identification of gaps and wishes, dialogue / forum in the plenary, choice of activities and establishment of the priorities and WP, functional mailbox /e-mails; NRLs are polled on wishes for agenda items which are taken into the plenaries. NRLs are given he floor for presentations and discussions, Achieved ex-post: all implemented
FF.NRL.4 Number of NRLs visited for training	Expected ex-ante: In the context of the WP 2015, the actions have foreseen 1) a questionnaire of root cause analysis, 2) dialogue with NRLs concerned 3) an option for visiting and/or be given a new spiked material to practice. A >10% improvement is expected from those actions Achieved ex-post: All implemented
FF.NRL.5 Number of workshops/trainings to be organised other than the annual workshop within the framework of EURL activity as described in the AWP (third countries involvement to be stated as laid out in AWP)	Expected ex-ante: one training workshop on new advances for safety assessment of materials. One forum on ceramics. Achieved ex-post: all implemented
FF.NRL.6	Expected ex-ante:

Attendance rate and number of positive satisfaction surveys above 85% received for such workshops	it is expected that more than 90% received survey will be positive surveys
	Achieved ex-post:
	achieved

FF.COM.1 Number of qualified staff in terms of preparedness with relevant completed training able to travel, to assist during outbreaks/ crisis situations and/or to engage the EURL on-site	Expected ex-ante:
	Permanent Staff dedicated to food contact for DG SANTE include 3 permanent researchers, and 2 permanent technical staff. Temporary staff for the EURL included 3-4 contracts. The permanent staff has 10 to 17 years for expertise on food contact and able to provide ad hoc contribution to DG SANTE but are not eligible to be covered under the EURL financial rules. 2 permanent staff are working specifically under accreditation 17025 for the qualification of the EURL and one EURL contractual staff also do so. The EURL also has a functional mailbox for all stakeholders with quality criteria established for promptness (15 days) and quality of replies (acknowledgement of the help by stakeholders).
	Achieved ex-post:
	all implemented
FF.COM.2 Adequacy of response to requests in terms of 1) content and 2) timely delivery*	Expected ex-ante:
	The JRC established its activity of food contact in direct support to DG SANTE in 1995. It responds to any ad-hoc request in its field in its capacity of support to EU policies of long standing expertise. JRC attends Member States SANTE meetings. It provides contributions where needed on either planned or ad-hoc basis. It also contributes to data and tools for EFSA where indicated in absence of conflict with its distribution of competences prioritising the service to DG SANTE. Currently the JRC drafts a technical guidelines in support of regulation on plastics 10/2011 (migration testing).
	Achieved ex-post:
	all implemented
**Adequacy in terms of timeline and quality and attendance to EFSA/ECDC/EMA meetings to be agreed upon with the lab in a quantitative manner.*	

FF.CEN.1 Provision of consultant expertise to FAO/WHO/OECD (independently of a mandate as FAO etc reference laboratory)	Expected ex-ante:
	not applicable
	Achieved ex-post:
	The mandate of the EURL-FCM implies also to carry out a mutual and reciprocal exchange of information with competent laboratories in third countries, to international institutes, or to CEN/ISO standardisation of analytical methods. The EURL implemented in 2014 a functional mailbox for all stakeholders and established quality criteria for promptness (15 days) and quality of replies (acknowledgement of the help by stakeholders). The EURL had the most a prominent place as highlight for visitors of the JRC and in light of the Universal expo 2015 (Silvia's lab was represented by a food contact lab at the JRC)
FF.CEN.2 Participation to/leadership for the CEN/ISO standardisation of analytical methods	Expected ex-ante:
	TC 194 (participation/chairing since 1996) plastics Participation in ISO TC 166 was established in 2015
	Achieved ex-post:
	Currently the JRC as EURL FCM is involved in TC 172- paper and board (participation since 2011); TC 194 (participation/chairing since 1996) plastics when it becomes active again. Participation in ISO TC 166 was received positively and was established in 2014. The first meeting took place in Paris on 26-27 January 2015 with a presentation of the JRC work on ceramics.

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JRC Mission

As the Commission's in-house science service, the Joint Research Centre's mission is to provide EU policies with independent, evidence-based scientific and technical support throughout the whole policy cycle.

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Serving society
Stimulating innovation
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